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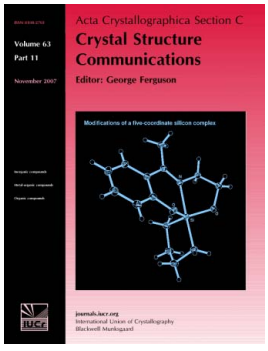

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Isolation, pharmacological activity and structure determination of physalin B and 5β,6β-epoxyphysalin B isolated from Congolese *Physalis angulata* L.

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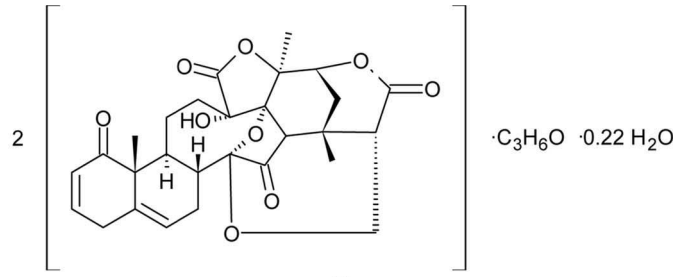
Physalis angulata L., an annual herb from the Solanaceae family, is widely used in popular medicine in tropical countries to treat a variety of diseases. Two products, (**X**) and (**Y**), were isolated from a crude CH_2Cl_2 extract of dried Congolese *Physalis angulata* L. plants and crystallized from acetone for structure elucidation. Compound (**X**) corresponds to a physalin B dimer acetone solvate hydrate ($2C_{28}H_{30}O_9\cdot C_3H_6O\cdot 0.22H_2O$), while compound (**Y**) crystallizes as a mixed crystal containing two physalin B molecules which overlap with 5β,6β-epoxyphysalin B, also known as physalin F, and one acetone molecule in the asymmetric unit ($1.332C_{28}H_{30}O_9\cdot 0.668C_{28}H_{30}O_{10}\cdot C_3H_6O$). Antiplasmodial activity, cytotoxic activity and selectivity indices were determined for crude extracts and the two isolated products (**X**) and (**Y**).

Keywords: crystal structure; *Physalis angulata* L.; physalin B; 5β,6β-epoxyphysalin B; antiplasmodial activity.

1. Introduction

In 1852, an amorphous bitter substance was isolated from the leaves of *Physalis alkekengi* and named physalin (Dessaigne & Chautard, 1852). In 1961, Völksen reported the isolation of a crystalline bitter compound from *Physalis franchettii*, a species very close to *Physalis alkekengi* (Völksen, 1961). A formula of $C_{21}H_6O_8$ was given for the bitter substance, but no further studies on its structure were reported. In 1969, the structure of a bitter substance isolated from *Physalis alkekengi* was reported as a steroid having an unusal 13,14-seco-16,24-cyclo steroidal-ring in which the C13—C14 bond is broken and replaced by a C16—C24 bond (Matsuura *et al.*, 1969). This seco-steroid with formula $C_{28}H_{30}O_{10}$ was named physalin A. Since then, more than a dozen physalins have been discovered and the structures of seven of them are deposited in the Cambridge Structural Database (CSD, Version 5.34; Allen, 2002). In physalin B, the O atom of the hydroxy group attached to C14 in physalin A is bridged to atom C27 (Matsuura *et al.*, 1970).



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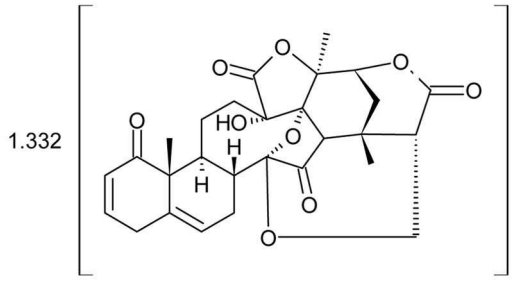

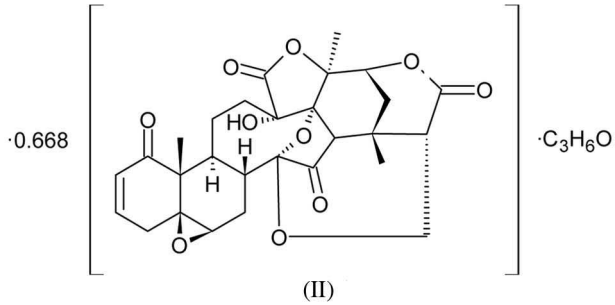

(II)

Plants of the gender *Physalis*, especially *Physalis angulata* L., are used in the folk medicine of many tropical countries, including African, American and Asian countries, as antimicrobial and antiparasitic agents (Mairura, 2008). In particular, physalin B and physalin F (5β,6β-epoxyphysalin B) are active against *Mycobacterium tuberculosis*, leukemia and malaria. They also act as immunosuppressive agents and have antihepatotoxic action (Sá *et al.*, 2011; Januário *et al.*, 2002; Chiang *et al.*, 1992).

Previously, we tested the antiplasmodial activity of crude extracts of *Physalis angulata* (CH_2Cl_2 and MeOH, H_2O and EtOH/H_2O) *in vitro* against the 3D7 and W2 (chloroquine-sensitive and chloroquine-resistant) strains of *Plasmodium falciparum* and *in vivo* in mice infected by the *Plasmodium berghei berghei* (Lusakibanza *et al.*, 2010). In the present study, we evaluate the *in vitro* antiplasmodial activity of crude extracts and of two isolated steroids physalin B and 5β,6β-

Table 1
Experimental details.

	(**X**)	(**Y**)
Crystal data		
Chemical formula	$2C_{28}H_{30}O_9\cdot C_3H_6O\cdot 0.22H_2O$	$1.332C_{28}H_{30}O_9\cdot 0.668C_{28}H_{30}O_{10}\cdot C_3H_6O$
M_r	1083.08	1089.81
Crystal system, space group	Monoclinic, $P2_1$	Monoclinic, $P2_1$
Temperature (K)	100	100
a, b, c (Å)	12.5153 (9), 14.1333 (11), 14.6716 (10)	12.4859 (4), 14.1716 (4), 14.6559 (7)
β (°)	96.863 (4)	96.829 (3)
V (Å^3)	2576.6 (3)	2574.89 (16)
Z	2	2
Radiation type	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	0.87	0.88
Crystal size (mm)	0.60 × 0.15 × 0.05	0.45 × 0.24 × 0.01
Data collection		
Diffractometer	Bruker SMART 6000 diffractometer	Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Absorption correction	Multi-scan (*SADABS*; Bruker, 2003)	Multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min}, T_{max}	0.624, 0.957	0.699, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25302, 9227, 7497	15953, 8953, 8208
R_{int}	0.082	0.054
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.615	0.595
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.147, 1.04	0.064, 0.175, 1.06
No. of reflections	9227	8953
No. of parameters	727	745
No. of restraints	4	50
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.38, −0.20	0.49, −0.32
Absolute structure	Flack (1983), 4126 Friedel pairs	Flack (1983), 4215 Friedel pairs
Absolute structure parameter	0.0 (2)	0.1 (2)

Computer programs: *SMART* (Bruker, 2003), *CrysAlis PRO* (Agilent, 2012), *SAINT* (Bruker, 2003), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

epoxyphysalin B from *Physalis angulata* L. against the 3D7 (chloroquine sensitive) strain of *Plasmodium falciparum*, together with the cytotoxic activity against the human normal foetal lung fibroblasts WI-38. This allowed the determination of the selectivity index after a phytochemical investigation of the CH_2Cl_2 extract. The structures of both isolated compounds were determined by X-ray crystallography.

2. Experimental

2.1. Isolation, crystallization and data collection

Physalis angulata L. plants were collected in the province of Bas-Congo (Democratic Republic of Congo) and identified at the Institut National pour l'Etudes et la Recherche Agronomiques de Kinshasa (INERA, University of Kinshasa). A voucher specimen for this plant was deposited in the herbarium of the institute and in the National Botanical Garden of Belgium (Meise, Belgium). Whole plants were air-dried at room temperature and powdered. A crude CH_2Cl_2 extract was obtained by maceration of 1 kg of powdered plants in 10 l for 30 min under constant stirring at room temperature. The extract was filtered and evaporated to dryness under reduced pressure at 313 K with a rotatory evaporator. Two products, (**X**) and (**Y**), were isolated by bio-guided fractionation of the crude extract using Si60 open-column chromatography (hexane/ethyl acetate as mobile phase, MeOH as eluent) followed by preparative high-performance liquid chromatography (HPLC) (RP-18 column in gradient mode with MeOH–H_2O = 70:30 *v*/*v*). The two compounds were separated by preparative thin-layer chromatography (TLC) with CH_2Cl_2–MeOH (95:5 *v*/*v*) as mobile phase, resulting in 30 mg of compound (**X**) and 10 mg of compound (**Y**). The NMR and MS data of (**X**) were in complete agreement with literature values (Bunsho *et al.*, 1995). The NMR data of (**Y**) were similar to those of (**X**). After structure elucidation by X-ray crystallography, the MS data of (**Y**) were recorded and showed the presence of two molecular ions, one corresponding to physalin B and a second corresponding to a supplementary O atom to the mass of physalin B. The powders of both samples were separately dissolved in acetone and plate-like crystals suitable for X-ray diffraction were obtained within 24 h by slow evaporation at room temperature. Crystal data and other data collection parameters for (**X**) and (**Y**) are given in Table 1.

2.2. Structure solution and refinement

H atoms of the water molecule were located in a difference map and refined further with AFIX 83 for H95*A* and DFIX commands O95—H95*B* = 0.84 Å and H95*A*—O95—H95*B* = 1.38 Å, with σ set at 0.02 Å. The other H atoms were posi-

Table 2
In vitro IC_{50} values (μg ml^{-1}) against *Plasmodium falciparum* (3D7) and against WI-38 cells, and selectivity index (SI).

Extracts	IC_{50} (3D7)	IC_{50} (WI-38)	SI
CH_2Cl_2	1.41±0.21	6.97±2.02	4.94
EtOH/H_2O (50:50 *v*/*v*)	9.05±0.87	36.48±4.41	4.02
H_2O	11.36±1.84	56.43±1.85	4.96
MeOH	3.16±1.13	13.95±3.68	4.41
(**X**)	0.86±0.13	3.13±0.81	3.63
(**Y**)†	0.62±0.35	1.59±0.27	2.25
Chloroquine	0.0022±0.005		ND
Artemisinin	0.0047±1.39		ND
Camptothecine		13.84±3.82	ND

† The activity of (**Y**) is in fact the activity of a (**Y1**)/(**Y2**) mixture.

Table 3
Hydrogen-bond geometry (Å, °) for (**X**).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O34—H34···O69	0.84	1.90	2.735 (3)	171
O74—H74···O29	0.84	2.02	2.806 (3)	156
O95—H95*A*···O37	0.84	1.71	2.513 (2)	161
O95—H95*B*···O74^{i}	0.86 (14)	2.12 (9)	2.924 (3)	155
C52—H52*A*···O76	0.99	2.40	3.232 (4)	141
C91—H91*B*···O36	0.98	2.59	3.456 (6)	147
C17—H17···O75^{ii}	1.00	2.59	3.421 (4)	141
C53—H53*B*···O77^{iii}	0.99	2.45	3.415 (5)	163
C59—H59···O94^{iv}	1.00	2.58	3.463 (5)	147
C63—H63···O77^{iii}	1.00	2.36	3.345 (5)	169

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+1$; (ii) $x+1, y, z$; (iii) $-x+1, y-\frac{1}{2}, -z+2$; (iv) $-x+1, y+\frac{1}{2}, -z+2$.

tioned with idealized geometry using a riding model, with C—H = 0.95–0.99 Å. All H atoms were refined with isotropic displacement parameters set at 1.2 or 1.5 times the U_{eq} of the parent atoms.

Both structures are disorderd. In the crystal of (**X**), water molecule O95 only partially occupies the site. The occupancy refined to 0.216 (11).

Crystal (**Y**) is a typical case of the cocrystallization of two compounds having very similar structures, differing only in the replacement of a double bond in the first compound (**Y1**) by an epoxide group in the second compound (**Y2**). The disorder was refined by applying the same displacement parameters for atoms C5*A*/C5*B*, C6*A*/C6*B*, C45*A*/C45*B* and C46*A*/C46*B* using the *SHELXTL* (Sheldrick, 2008) command EADP. Then the occupancies of disordered atoms related to the compound (**Y1**) were set to free variable 21 in molecule I and to 31 in molecule II. Those of compound (**Y2**), including O39 and O79, were set at −21 and −31, respectively. For molecule I, the following occupancies were obtained: 0.761 (14) for compound (**Y1**) and 0.239 (14) for compound (**Y2**); for molecule II: 0.570 (13) for (**Y1**) and 0.430 (13) for (**Y2**). The distances between disordered atoms were restrained using DFIX and SADI commands with σ set to 0.02 Å (C5*A*—C6*A* = C45*A*—C46*A* = 1.30 Å, C5*B*—C6*B* = C45*B*—C46*B* = 1.47 Å, C10—C5*A* = C10—C5*B* = C50—C45*B* = C50—C45*A* = 1.53 Å, C6*A*—C7 = C46*A*—C47 = 1.48 Å, C6*B*—C7 = C46*B*—C47 = 1.50 Å, and C5*A*—C4 = C45*A*—C44 = C5*B*—C4 = C45*B*—C44 = 1.50 Å).

The absolute configuration of (**X**) was determined on the basis of the Flack and Hooft parameters of 0.12 (18) and −0.02 (17), respectively (Hooft *et al.*, 2008). For (**Y**), a Flack parameter of 0.1 (2) and a Hooft parameter of 0.04 (10) were obtained.

Structure refinement details are summarized in Table 1.

2.3. *In vitro* antiplasmodial assay

The culture of *Plasmodium falciparum* strain was carried out as described previously (Frédérich *et al.*, 2001). All crude extracts and pure compounds were evaluated *in vitro* for their activity against a chloroquine-sensitive strain of *Plasmodium falciparum* (3D7). For each crude (extract and pure) compound, a series of eight threefold dilutions (from 200 to 0.09 g ml^{-1}) was prepared, placed in two rows of a 96-well microplate and tested in triplicate. Artemisinin (98%, Sigma–Aldrich) and chloroquine diphosphate salt (Sigma–Aldrich) were used as standards, and infected and uninfected erythrocytes were added as positive and negative controls, respectively. After 48 h of incubation at 310 K, the level of parasitaemia was estimated by measuring lactate dehydrogenase activity, as described previously (Jonville *et al.*, 2008; Lusakibanza *et al.*, 2010). The results were expressed as the mean IC_{50} (the concentration of a drug that reduced the level of parasitaemia to 50%) and shown in Table 2.

2.4. *In vitro* cytotoxic assay

Cells from the human normal foetal lung fibroblast cell line, WI-38, were cultivated *in vitro* in DMEM (Dubecco's modified Eagle's medium, Lonzo, Belgium) and incubated at 310 K in a humidified atmosphere with 5% CO_2. For each sample, six threefold dilutions (from 200 to 0.82 g ml^{-1}) were prepared, placed in three rows of a 96-well microplate and tested at least twice. Camptothecin (Sigma) was used as a positive control. After 48 h incubation, cell viability was determined by measuring the fibroblast mitochondrial enzyme activity, as described previously (Stevigny *et al.*, 2002). The results were

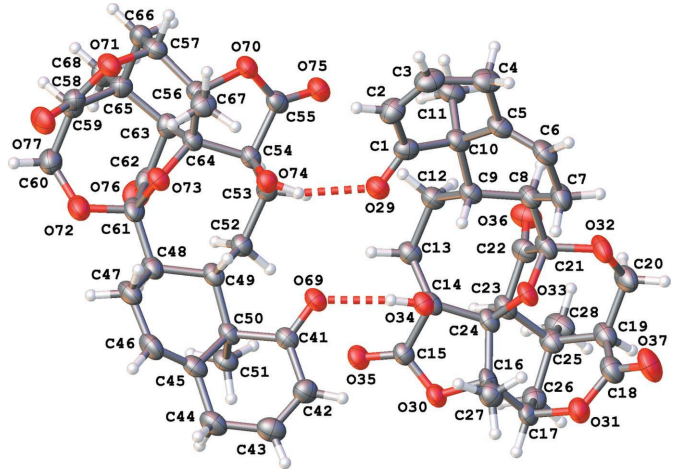

Figure 1
The molecular structure of compound (**X**), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Acetone and water molecules have been omitted for clarity.

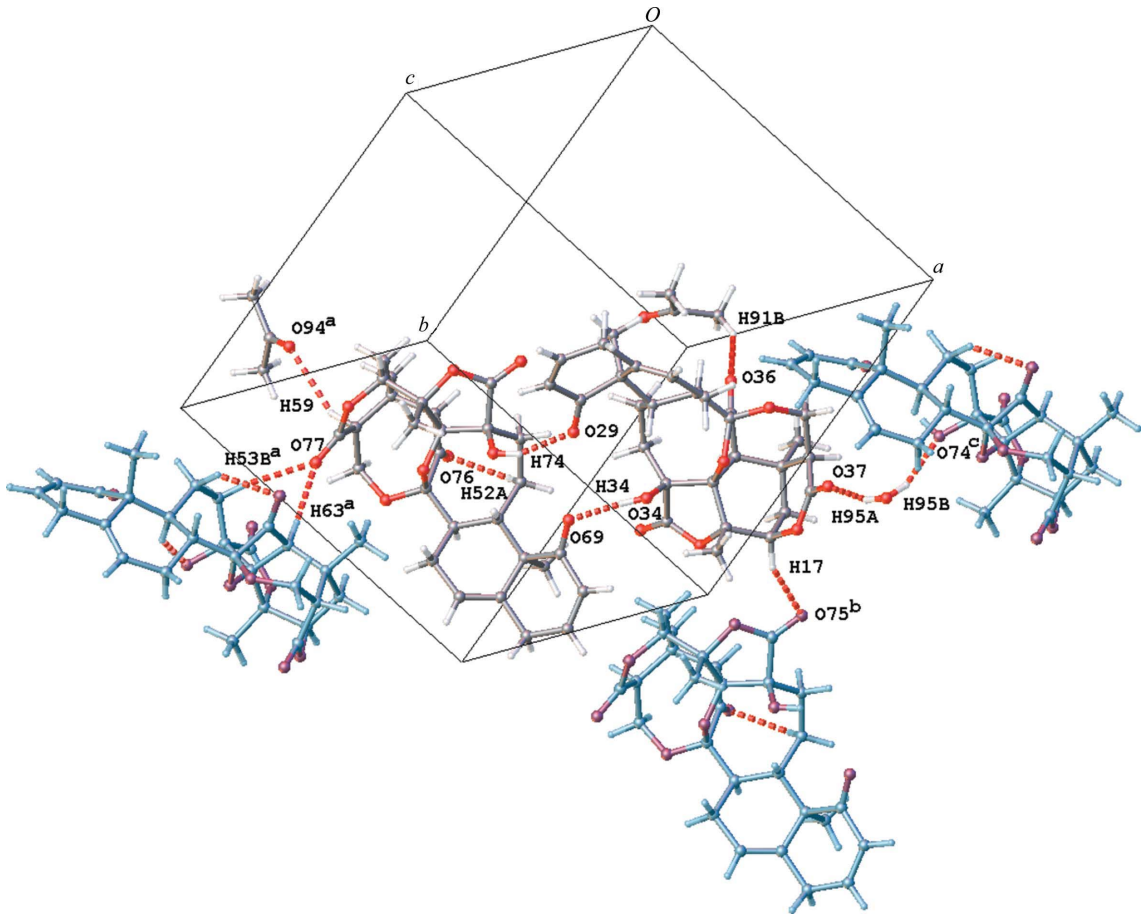

Figure 2
The hydrogen-bonding interactions (dashed lines) in the crystal packing of (**X**). Symmetry-related physalin molecules are on the left, right and bottom (shown in blue in the electronic version of the paper). [Symmetry codes: (*a*) $-x+1, y+\frac{1}{2}, -z+2$; (*b*) $x+1, y, z$; (*c*) $-x+2, y-\frac{1}{2}, -z+1$.]

expressed by the mean of IC_{50} of at least two independent assays and the selectivity index [the ratio between the cytotoxic (WI-38 cells) and antiparasitic (3D7 strain) activity] was calculated and shown in Table 2.

3. Results and discussion

The structure of compound (**X**) corresponds to physalin B acetone hemisolvate 0.11-hydrate ($2C_{28}H_{30}O_9\cdot C_3H_6O\cdot 0.22H_2O$). The asymmetric unit consists of two physalin B molecules, known as (8*R*,9*S*,10*R*,13*S*,14*R*,16*S*,17*R*,20*S*,22*R*,24*R*,25*S*)-16,24-cyclo-13,14-secoergosta-2,5-diene-1,15-dioxo-14:17,14:27-diepoxy-13-hydroxy-18,26-dioic acid 18:20,26:22-di-γ-lactone δ-lactone, one water molecule with an occupancy of 0.22 and one acetone molecule. A least-squares fit (r.m.s. deviation of fitted atoms = 0.057 Å, maximal deviation 0.157 Å) indicates the similarities between the two molecules. The physalin B molecules in the asymmetric unit are strongly hydrogen bonded through two O—H···O contacts (O34—H34···O69 and O74—H74···O29; Table 3) creating a dimer. The non-H atom-numbering ranges from 1 to 37 (molecule I), from 41 to 77 (molecule II) and from 91 to 95 (solvent) (Fig. 1).

Compound (**X**) is a highly oxygenated steroidal lactone containing eight fused rings. Due to the strong similarity between both molecules, numerical values are only given for molecule I. The six-membered rings *A* (atoms C1–C5/C10) and *B* (C5–C10), which is *trans*-fused to ring *A*, are in half-chair conformations, with atoms C5 and C10 deviating from the best plane through atoms C1/C2/C3/C4 by −0.164 and 0.488 Å, respectively, and atoms C7 and C8 deviating from the best plane through C5/C6/C10/C9 by 0.212 and 0.795 Å, respectively. The two spiro-fused five-membered rings *D* (O30/C14–C16/C24) and *E* (O33/C21–C24) adopt envelope (on C24) and half-chair conformations, respectively; in ring *D*, atom C24 deviates by 0.618 Å from the best plane through atoms O3O/C15/C14/C16 and in ring *E* the deviations of atoms C22 and C24 from the best plane through atoms O33/C21/C23 are 0.225 and 0.445 Å, respectively. Rings *F* (C16–C17/C23–C26) and *G* (O31/C17–C19/C25–C26) are in a chair conformation. The two epoxy seven- and eight-membered rings *H* (O32/C19–C23/C25) and *C* (C8–C9/C12–C14/C21/C24/O33) are in chair and boat–chair conformations, respectively.

The packing of compound (**X**) is characterized by a network of hydrogen bonds, directly between the physalin B dimers or through water and/or acetone molecules (Table 3 and Fig. 2). The construction of the network in the crystallographic *b* direction is essentially ensured through O—H···O interactions with water molecules. In the crystallographic *a* direction, the network is formed by a C17—H17···O75^{ii} contact. In the crystallographic *c* direction, dimers are linked through C—

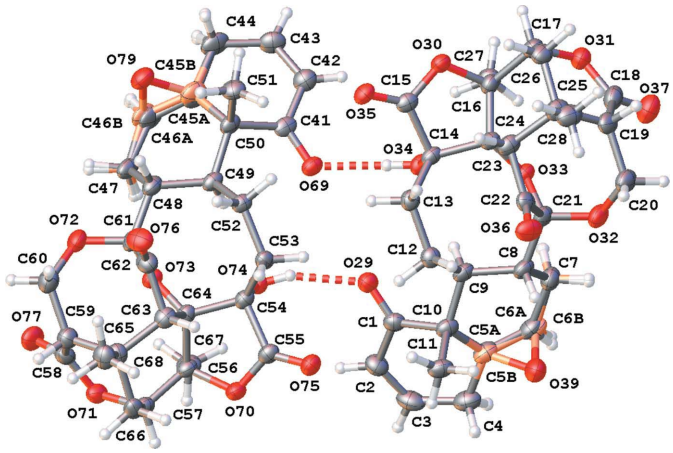

Figure 3
The molecular structure of compound (**Y**), showing the atom-numbering scheme. The 5β,6β-epoxy fragment is shown in orange in the electronic version of the paper. Displacement ellipsoids are drawn at the 50% probability level. The acetone molecule has been omitted for clarity.

H···O hydrogen bonds involving acetone molecules. The network is further strengthened by bifurcated C53—H53*B*···O77^{iii} and C63—H63···O77^{iii} hydrogen bonds.

The asymmetric unit of compound (**Y**) contains two physalin B-like molecules and an acetone molecule. It became clear from difference electron-density maps that each independent molecular site in the structure is in fact occupied by a mixture of physalin B [refered to as (**Y1**)] and 5β,6β-epoxyphysalin B [refered to as (**Y2**)]. A least-squares fit (r.m.s. deviation fitted atoms = 0.053 Å) indicates a similar conformation and configuration for the two molecules. As in the crystal of (**X**), the physalin molecules in the asymmetric unit of (**Y**) form a dimer *via* O—H···O contacts (O34—H34···O69 and O74—H74···O29; Table 4). Non-H atoms are numbered in the same way as in compound (**X**), with in addition atom O39 in molecule I and O79 in molecule II; C5*A* and C6*A* belong to (**Y1**), whereas C5*B* and C6*B* belong to (**Y2**) in

Table 4
Hydrogen-bond geometry (Å, °) for (**Y**).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O34—H34···O69	0.84	1.91	2.739 (4)	172
O74—H74···O29	0.84	2.00	2.801 (4)	160
C52—H52*A*···O76	0.99	2.38	3.217 (5)	142
C17—H17···O75^{i}	1.00	2.59	3.415 (5)	140
C53—H53*B*···O77^{ii}	0.99	2.49	3.453 (5)	163
C63—H63···O77^{ii}	1.00	2.39	3.376 (5)	170
C66—H66*B*···O94^{iii}	0.99	2.58	3.457 (6)	148
C91—H91*C*···O37^{iv}	0.98	2.58	3.432 (7)	145

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, y-\frac{1}{2}, -z+2$; (iii) $-x+1, y+\frac{1}{2}, -z+2$; (iv) $-x+2, y-\frac{1}{2}, -z+1$.

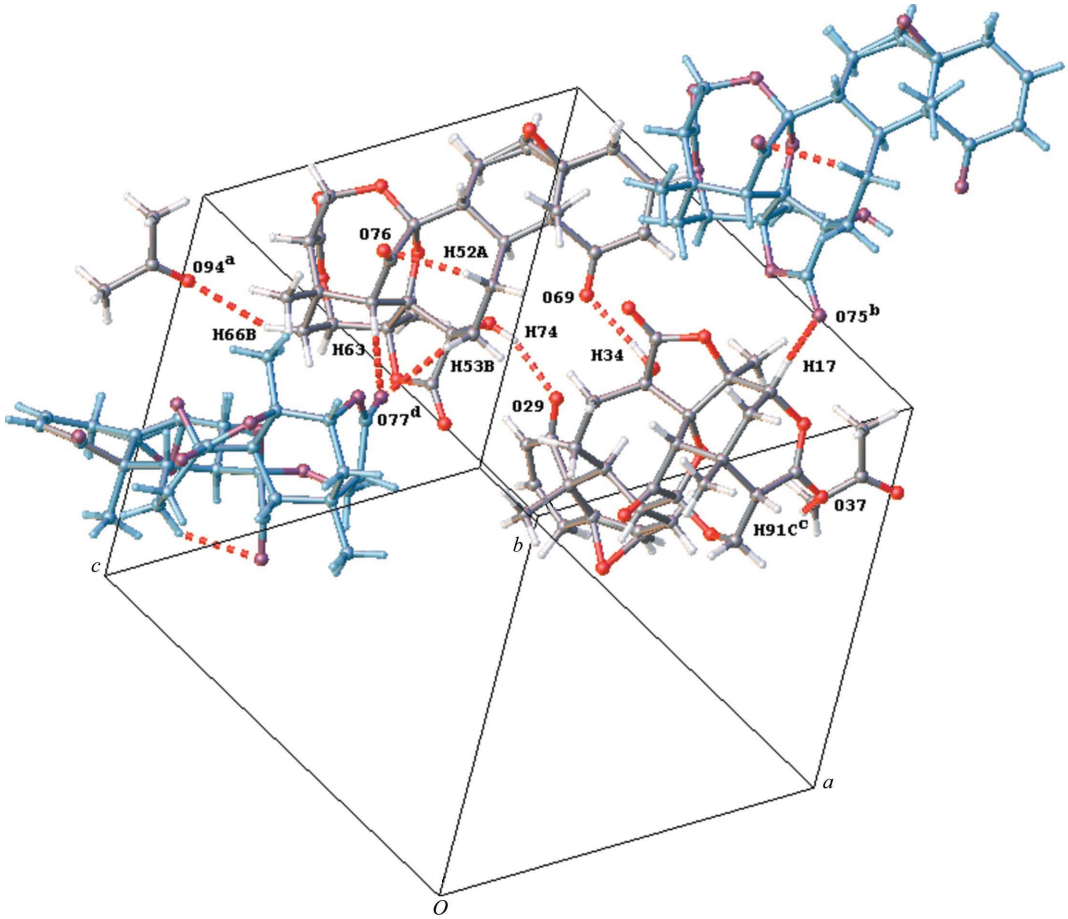

Figure 4
The hydrogen-bonding interactions (dashed lines) in the packing of (**Y**). Symmetry-related physalin molecules are on the left and right (shown in blue in the electronic version of the paper). [Symmetry codes: (*a*) $-x+1, y+\frac{1}{2}, -z+2$; (*b*) $x+1, y, z$; (*c*) $-x+2, y+\frac{1}{2}, -z+1$; (*d*) $-x+1, y-\frac{1}{2}, -z+2$.]

molecule I and C45*A* and C46*A* belong to (**Y1**), whereas C45*B* and C46*B* belong to (**Y2**) in molecule II (Fig. 3).

Analyzing separately the structures of (**Y1**) and (**Y2**) shows that the ring conformations and substituent orientations in compounds (**Y1**) and (**Y2**) are similar to compound (**X**), and (**Y2**) differs from (**Y1**) [or (**X**)] only by its extra-three-membered epoxide group O39/C5*B*/C6*B*, with C5*B*—O39 and C6*B*—O39 bonds in an axial orientation. The least-squares fits of both molecules I and II of compounds (**Y1**) and (**Y2**) (r.m.s. deviation fitted atoms = 0.052 and 0.051 Å, respectively) indicate similarities between molecules I and II. A least-squares fit of (**Y1**) and (**X**) gives an r.m.s. deviation of the fitted atoms of 0.016 Å and a maximal deviation of 0.032 Å.

The packing of (**Y**) is characterized by a network of hydrogen bonds involving the physalin dimers and acetone molecule (Table 4 and Fig. 4). In the crystallographic *a* direction, the network growth is ensured by means of the C17—H17$\cdots$O75^{i} interaction. In the *b* direction, dimers are directly connected through C53—H53*B*$\cdots$O77^{ii} and C63—H63$\cdots$O77^{ii} bifurcated hydrogen bonds. In the *c* direction, dimers are linked through the acetone molecule by means of hydrogen bonds.

Compared to 5α,6α-epoxyphysalin B (Kawai *et al.*, 1994), (**Y2**) or 5β,6β-epoxyphysalin B differs only by the epoxide group orientation. The asymmetric unit of 5α,6α-epoxyphysalin B comprises only one physalin molecule, resulting in different unit-cell parameters. A strong intramolecular O34—H34$\cdots$O69 hydrogen bond prevents the formation of dimers.

Seven physalin structures are deposited in the Cambridge Structural Database (CSD, Version 5.34; Allen, 2002). None of these physalins crystallizes as a dimer as observed for (**X**) and (**Y**). Instead, intramolecular hydrogen bonds are present, or hydrogen bonds with surrounding solvent molecules, preventing the formation of dimers.

The two fractions (**X**) and (**Y**) isolated from *Physalis angulata* showed the highest activity against the chloroquine-sensitive strain of *Plasmodium falciparum* with an IC_{50} < 1 µg ml^{-1} (Table 2). The results confirmed the activity of some extracts of *Physalis angulata* described previously (Muregi *et al.*, 2004; Kvist *et al.*, 2006; Ankrah *et al.*, 2003) and of physalins B, F, G and D (Sá *et al.*, 2011).

The cytotoxic assay was performed on human lung fibroblasts (WI-38). The selectivity index SI is defined as the ratio of the cytotoxic IC_{50} value and the parasitic IC_{50} value. As described previously (Zirihi *et al.*, 2005; Lusakibanza *et al.*, 2010), the cytotoxicity of *Physalis angulata* extracts is caused by physalin B and 5β-6β-epoxyphysalin B. The two isolated compounds show an SI between 2 and 4 (Table 2).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG3314). Services for accessing these data are described at the back of the journal.

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supplementary materials

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Isolation, pharmacological activity and structure determination of physalin B and 5*β*,6*β*-epoxyphysalin B isolated from Congolese *Physalis angulata* L.

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Computing details

Data collection: *SMART* (Bruker, 2003) for (X); *CrysAlis PRO* (Agilent, 2012) for Y. Cell refinement: *SAINT* (Bruker, 2003) for (X); *CrysAlis PRO* (Agilent, 2012) for Y. Data reduction: *SAINT* (Bruker, 2003) for (X); *CrysAlis PRO* (Agilent, 2012) for Y. For both compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

(X) (8*R*,9*S*,10*R*,13*S*,14*R*,16*S*,17*R*,20*S*,22*R*,24*R*,25*S*)-16,24-Cyclo-13,14-secoergosta-2,5-diene-1,15-dioxo-14:17,14:27-diepoxy-13-hydroxy-18,26-dioic acid 18:20,26:22-di-*γ*-lactone *δ*-lactone acetone hemisolvate 0.11-hydrate

Crystal data

$2C_{28}H_{30}O_9 \cdot C_3H_6O \cdot 0.22(H_2O)$
$M_r = 1083.08$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 12.5153$ (9) Å
$b = 14.1333$ (11) Å
$c = 14.6716$ (10) Å
$\beta = 96.863$ (4)°
$V = 2576.6$ (3) Å^3
$Z = 2$
$F(000) = 1148.4$
$D_x = 1.396$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
$\mu = 0.87$ mm^{-1}
$T = 100$ K
Plate, colorless
0.60 × 0.15 × 0.05 mm

Data collection

Bruker SMART 6000 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min} = 0.624$, $T_{max} = 0.957$
25302 measured reflections
9227 independent reflections
7497 reflections with $I > 2\sigma(I)$
$R_{int} = 0.082$
$\theta_{max} = 71.6°$, $\theta_{min} = 3.0°$
$h = -15 \rightarrow 15$
$k = -17 \rightarrow 17$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.147$
$S = 1.04$
9227 reflections
727 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983), 4126 Friedel pairs
Absolute structure parameter: 0.0 (2)

Special details

Experimental. For compound (**X**), diffraction data were collected on a Bruker diffractometer equipped with a *SMART* 6000 CCD detector and copper X-ray tube with crossed Göbel mirrors. A dataset of 4602 frames having a width of 0.3° for low and intermediate angles, and 0.5° for high angles was collected to a θ-value of 66.59° corresponding to a resolution of 0.840?Å [(Bruker, 2003). Data were integrated by the *SAINT* program (Bruker, 2003) and a multi-scan absorption correction was performed by the program *SADABS* (Bruker, 2003) leading to Tmin and Tmax of 0.624 and 0957, respectively. The crystal belongs to the monoclinic space group P21 with R_{int}-value of 8.20% and a completeness of 99.8%.
The structure was solved by direct methods using the *SHELX* program and refined according to the least-quares methods by *SHELXTL* package (Bruker, 2003). Non-hydrogen atoms were located in a fourrier map and refined anisotropically.
Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.6692 (3)	0.5254 (3)	0.5749 (2)	0.0371 (8)	
C2	0.5643 (3)	0.5674 (3)	0.5419 (3)	0.0446 (9)	
H2	0.5316	0.6102	0.5801	0.054*	
C3	0.5144 (3)	0.5460 (3)	0.4591 (3)	0.0498 (10)	
H3	0.4481	0.5765	0.4393	0.060*	
C4	0.5572 (3)	0.4767 (3)	0.3957 (3)	0.0482 (10)	
H4A	0.5533	0.5051	0.3337	0.058*	
H4B	0.5109	0.4197	0.3912	0.058*	
C5	0.6730 (3)	0.4471 (3)	0.4265 (2)	0.0401 (8)	
C6	0.7433 (3)	0.4354 (3)	0.3678 (2)	0.0449 (9)	
H6	0.7202	0.4475	0.3049	0.054*	
C7	0.8568 (3)	0.4045 (3)	0.3932 (2)	0.0455 (9)	
H7A	0.9049	0.4602	0.3940	0.055*	
H7B	0.8771	0.3599	0.3462	0.055*	
C8	0.8726 (3)	0.3562 (3)	0.4878 (2)	0.0383 (8)	
H8	0.8330	0.2946	0.4831	0.046*	

C9	0.8247 (3)	0.4184 (3)	0.5581 (2)	0.0344 (7)
H9	0.8594	0.4819	0.5559	0.041*
C10	0.7016 (3)	0.4334 (3)	0.5298 (2)	0.0372 (8)
C11	0.6321 (3)	0.3512 (3)	0.5604 (3)	0.0441 (9)
H11A	0.6536	0.2919	0.5333	0.066*
H11B	0.5560	0.3637	0.5397	0.066*
H11C	0.6426	0.3461	0.6274	0.066*
C12	0.8444 (3)	0.3843 (3)	0.6597 (2)	0.0360 (7)
H12A	0.8492	0.3144	0.6586	0.043*
H12B	0.7792	0.4004	0.6886	0.043*
C13	0.9433 (3)	0.4210 (3)	0.7246 (2)	0.0357 (7)
H13A	0.9155	0.4618	0.7713	0.043*
H13B	0.9772	0.3654	0.7575	0.043*
C14	1.0317 (3)	0.4754 (2)	0.6857 (2)	0.0331 (7)
C15	1.1233 (3)	0.5051 (2)	0.7611 (2)	0.0322 (7)
C16	1.2040 (3)	0.4891 (3)	0.6277 (2)	0.0350 (8)
C17	1.3079 (3)	0.4373 (3)	0.6126 (2)	0.0361 (8)
H17	1.3701	0.4766	0.6397	0.043*
C18	1.2608 (3)	0.3619 (3)	0.4639 (3)	0.0470 (9)
C19	1.2247 (3)	0.2743 (3)	0.5100 (3)	0.0439 (9)
H19	1.2830	0.2278	0.5019	0.053*
C20	1.1251 (3)	0.2308 (3)	0.4525 (3)	0.0487 (10)
H20A	1.1465	0.2121	0.3922	0.058*
H20B	1.1058	0.1721	0.4836	0.058*
C21	0.9936 (3)	0.3354 (3)	0.5114 (2)	0.0390 (8)
C22	1.0246 (3)	0.2764 (3)	0.5983 (3)	0.0388 (8)
C23	1.1229 (3)	0.3229 (3)	0.6489 (2)	0.0375 (8)
H23	1.1233	0.3154	0.7167	0.045*
C24	1.1019 (3)	0.4268 (3)	0.6203 (2)	0.0333 (7)
C25	1.2253 (3)	0.2780 (3)	0.6170 (3)	0.0410 (8)
C26	1.3194 (3)	0.3406 (3)	0.6551 (2)	0.0414 (8)
H26A	1.3213	0.3460	0.7226	0.050*
H26B	1.3878	0.3116	0.6417	0.050*
C27	1.1953 (3)	0.5798 (3)	0.5718 (2)	0.0416 (8)
H27A	1.2664	0.6096	0.5746	0.062*
H27B	1.1452	0.6233	0.5969	0.062*
H27C	1.1688	0.5652	0.5078	0.062*
C28	1.2386 (3)	0.1792 (3)	0.6572 (3)	0.0506 (10)
H28A	1.1776	0.1398	0.6322	0.076*
H28B	1.2412	0.1826	0.7241	0.076*
H28C	1.3056	0.1514	0.6411	0.076*
C41	0.9749 (3)	0.7110 (2)	0.8356 (2)	0.0328 (7)
C42	1.0719 (3)	0.7573 (3)	0.8106 (3)	0.0412 (8)
H42	1.1043	0.7344	0.7595	0.049*
C43	1.1158 (3)	0.8310 (3)	0.8581 (3)	0.0463 (9)
H43	1.1777	0.8597	0.8384	0.056*
C44	1.0734 (3)	0.8702 (3)	0.9392 (3)	0.0458 (9)
H44A	1.0679	0.9398	0.9324	0.055*
H44B	1.1254	0.8568	0.9940	0.055*

C45	0.9643 (3)	0.8318 (3)	0.9558 (2)	0.0374 (8)
C46	0.8889 (3)	0.8843 (3)	0.9887 (3)	0.0446 (9)
H46	0.9035	0.9498	0.9980	0.053*
C47	0.7844 (3)	0.8486 (3)	1.0117 (3)	0.0433 (9)
H47A	0.7269	0.8683	0.9632	0.052*
H47B	0.7690	0.8776	1.0702	0.052*
C48	0.7822 (3)	0.7408 (3)	1.0211 (2)	0.0386 (8)
H48	0.8294	0.7229	1.0784	0.046*
C49	0.8292 (3)	0.6956 (2)	0.9381 (2)	0.0337 (7)
H49	0.7860	0.7203	0.8813	0.040*
C50	0.9462 (3)	0.7267 (2)	0.9343 (2)	0.0319 (7)
C51	1.0294 (3)	0.6707 (3)	1.0016 (2)	0.0387 (8)
H51A	1.0076	0.6726	1.0635	0.058*
H51B	1.1007	0.6995	1.0023	0.058*
H51C	1.0321	0.6048	0.9812	0.058*
C52	0.8203 (3)	0.5864 (3)	0.9348 (2)	0.0340 (7)
H52A	0.8174	0.5643	0.9984	0.041*
H52B	0.8883	0.5620	0.9155	0.041*
C53	0.7262 (3)	0.5374 (3)	0.8733 (2)	0.0345 (7)
H53A	0.7559	0.5118	0.8187	0.041*
H53B	0.7028	0.4826	0.9080	0.041*
C54	0.6265 (3)	0.5944 (2)	0.8393 (2)	0.0324 (7)
C55	0.5400 (3)	0.5307 (3)	0.7861 (2)	0.0351 (8)
C56	0.4475 (3)	0.6536 (3)	0.8486 (2)	0.0359 (7)
C57	0.3489 (3)	0.6469 (3)	0.9018 (2)	0.0378 (8)
H57	0.2844	0.6346	0.8563	0.045*
C58	0.3932 (3)	0.7622 (3)	1.0228 (2)	0.0377 (8)
C59	0.4431 (3)	0.6849 (3)	1.0851 (2)	0.0375 (8)
H59	0.3890	0.6752	1.1291	0.045*
C60	0.5396 (3)	0.7254 (3)	1.1455 (3)	0.0464 (9)
H60A	0.5131	0.7763	1.1833	0.056*
H60B	0.5689	0.6749	1.1881	0.056*
C61	0.6670 (3)	0.7098 (3)	1.0315 (2)	0.0387 (8)
C62	0.6505 (3)	0.6057 (3)	1.0538 (2)	0.0386 (8)
C63	0.5532 (3)	0.5721 (3)	0.9917 (2)	0.0371 (8)
H63	0.5616	0.5046	0.9736	0.045*
C64	0.5582 (3)	0.6392 (3)	0.9085 (2)	0.0312 (7)
C65	0.4538 (3)	0.5846 (3)	1.0430 (2)	0.0368 (8)
C66	0.3544 (3)	0.5705 (3)	0.9727 (2)	0.0385 (8)
H66A	0.3581	0.5080	0.9429	0.046*
H66B	0.2886	0.5720	1.0042	0.046*
C67	0.4379 (3)	0.7405 (3)	0.7883 (2)	0.0422 (9)
H67A	0.3660	0.7425	0.7534	0.063*
H67B	0.4924	0.7381	0.7456	0.063*
H67C	0.4489	0.7972	0.8266	0.063*
C68	0.4563 (3)	0.5089 (3)	1.1173 (2)	0.0460 (9)
H68A	0.5212	0.5168	1.1610	0.069*
H68B	0.4568	0.4461	1.0890	0.069*
H68C	0.3925	0.5152	1.1495	0.069*

C91	0.9135 (4)	0.0213 (4)	0.7558 (3)	0.0649 (13)	
H91A	0.9558	0.0125	0.8158	0.097*	
H91B	0.9574	0.0538	0.7144	0.097*	
H91C	0.8911	−0.0405	0.7299	0.097*	
C92	0.8163 (4)	0.0794 (4)	0.7667 (3)	0.0548 (11)	
C93	0.7456 (4)	0.1055 (4)	0.6834 (3)	0.0562 (11)	
H93A	0.6879	0.1467	0.6998	0.084*	
H93B	0.7142	0.0482	0.6536	0.084*	
H93C	0.7874	0.1391	0.6412	0.084*	
O29	0.7297 (2)	0.56541 (18)	0.63524 (16)	0.0398 (6)	
O30	1.21664 (19)	0.51225 (18)	0.72622 (14)	0.0359 (5)	
O31	1.3116 (2)	0.4317 (2)	0.51370 (15)	0.0417 (6)	
O32	1.0298 (2)	0.2884 (2)	0.43611 (17)	0.0474 (7)	
O33	1.0536 (2)	0.42148 (18)	0.52667 (14)	0.0364 (6)	
O34	0.99274 (19)	0.56079 (16)	0.64335 (14)	0.0326 (5)	
H34	0.9638	0.5936	0.6813	0.049*	
O35	1.1145 (2)	0.52265 (19)	0.84060 (14)	0.0375 (6)	
O36	0.9827 (2)	0.20308 (19)	0.6166 (2)	0.0505 (7)	
O37	1.2549 (3)	0.3688 (2)	0.38115 (18)	0.0618 (9)	
O69	0.91786 (19)	0.66204 (18)	0.78141 (14)	0.0353 (5)	
O70	0.44170 (19)	0.56874 (19)	0.79062 (15)	0.0397 (6)	
O71	0.3349 (2)	0.73799 (18)	0.94263 (16)	0.0387 (6)	
O72	0.6284 (2)	0.7637 (2)	1.10110 (17)	0.0462 (6)	
O73	0.59737 (19)	0.72647 (18)	0.94705 (14)	0.0346 (5)	
O74	0.64956 (19)	0.66668 (17)	0.77693 (15)	0.0342 (5)	
H74	0.6903	0.6450	0.7404	0.051*	
O75	0.5523 (2)	0.46110 (19)	0.74246 (17)	0.0420 (6)	
O76	0.7004 (2)	0.5622 (2)	1.11665 (16)	0.0496 (7)	
O77	0.3952 (2)	0.8439 (2)	1.04495 (19)	0.0484 (6)	
O94	0.7941 (3)	0.1042 (4)	0.8426 (2)	0.0892 (14)	
O95	1.3355 (14)	0.3725 (10)	0.2326 (9)	0.062 (6)	0.216 (11)
H95A	1.3174	0.3818	0.2852	0.093*	0.216 (11)
H95B	1.36 (2)	0.316 (6)	0.228 (8)	0.093*	0.216 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0347 (19)	0.038 (2)	0.0374 (17)	−0.0037 (14)	0.0013 (14)	0.0063 (16)
C2	0.040 (2)	0.047 (2)	0.046 (2)	0.0084 (16)	0.0040 (16)	0.0046 (18)
C3	0.033 (2)	0.063 (3)	0.051 (2)	0.0035 (17)	−0.0038 (16)	0.011 (2)
C4	0.042 (2)	0.058 (3)	0.0416 (19)	−0.0038 (18)	−0.0096 (16)	0.0057 (19)
C5	0.041 (2)	0.039 (2)	0.0365 (17)	−0.0030 (15)	−0.0095 (15)	−0.0018 (16)
C6	0.050 (2)	0.045 (2)	0.0365 (18)	−0.0003 (17)	−0.0106 (16)	−0.0075 (17)
C7	0.050 (2)	0.051 (2)	0.0334 (17)	0.0091 (18)	−0.0041 (15)	−0.0109 (17)
C8	0.039 (2)	0.0355 (19)	0.0379 (17)	0.0022 (14)	−0.0045 (14)	−0.0082 (15)
C9	0.0372 (19)	0.0327 (19)	0.0319 (15)	−0.0025 (14)	−0.0024 (13)	−0.0001 (14)
C10	0.039 (2)	0.039 (2)	0.0318 (16)	−0.0027 (15)	−0.0039 (13)	0.0006 (15)
C11	0.041 (2)	0.044 (2)	0.0438 (19)	−0.0057 (16)	−0.0080 (16)	0.0006 (18)
C12	0.038 (2)	0.0330 (18)	0.0359 (17)	−0.0015 (14)	−0.0011 (14)	0.0001 (15)

C13	0.0404 (19)	0.0329 (18)	0.0322 (15)	−0.0004 (14)	−0.0027 (13)	0.0018 (15)
C14	0.0410 (19)	0.0290 (18)	0.0265 (15)	0.0023 (13)	−0.0076 (13)	−0.0025 (14)
C15	0.0371 (19)	0.0304 (18)	0.0279 (16)	0.0000 (13)	−0.0006 (13)	−0.0003 (14)
C16	0.045 (2)	0.039 (2)	0.0200 (14)	0.0007 (15)	−0.0017 (13)	−0.0062 (14)
C17	0.0380 (19)	0.046 (2)	0.0230 (14)	−0.0013 (15)	−0.0006 (13)	−0.0060 (15)
C18	0.052 (2)	0.049 (2)	0.0380 (19)	0.0038 (17)	−0.0051 (16)	−0.0081 (18)
C19	0.044 (2)	0.042 (2)	0.0435 (19)	0.0083 (16)	−0.0053 (16)	−0.0102 (17)
C20	0.046 (2)	0.044 (2)	0.054 (2)	0.0099 (17)	−0.0038 (17)	−0.0177 (19)
C21	0.044 (2)	0.0313 (19)	0.0391 (18)	0.0056 (15)	−0.0070 (15)	−0.0108 (16)
C22	0.042 (2)	0.0257 (19)	0.047 (2)	0.0026 (14)	−0.0016 (15)	−0.0030 (16)
C23	0.044 (2)	0.0316 (19)	0.0347 (17)	0.0018 (15)	−0.0055 (15)	−0.0054 (15)
C24	0.0407 (19)	0.0332 (18)	0.0236 (14)	0.0026 (14)	−0.0058 (12)	−0.0023 (14)
C25	0.038 (2)	0.040 (2)	0.0426 (19)	0.0083 (15)	−0.0054 (15)	−0.0039 (17)
C26	0.037 (2)	0.047 (2)	0.0379 (18)	0.0065 (16)	−0.0037 (14)	−0.0025 (17)
C27	0.046 (2)	0.043 (2)	0.0352 (17)	−0.0019 (16)	0.0020 (15)	0.0013 (17)
C28	0.042 (2)	0.040 (2)	0.066 (3)	0.0084 (16)	−0.0115 (18)	−0.0046 (19)
C41	0.0369 (19)	0.0272 (17)	0.0322 (16)	0.0008 (13)	−0.0036 (13)	0.0017 (14)
C42	0.043 (2)	0.041 (2)	0.0388 (18)	−0.0030 (15)	0.0049 (15)	0.0033 (16)
C43	0.048 (2)	0.040 (2)	0.048 (2)	−0.0061 (17)	−0.0029 (17)	0.0070 (18)
C44	0.052 (2)	0.035 (2)	0.048 (2)	−0.0065 (16)	−0.0054 (17)	−0.0040 (17)
C45	0.041 (2)	0.034 (2)	0.0341 (17)	−0.0030 (15)	−0.0093 (14)	−0.0012 (15)
C46	0.052 (2)	0.033 (2)	0.046 (2)	0.0026 (16)	−0.0066 (16)	−0.0118 (17)
C47	0.042 (2)	0.038 (2)	0.047 (2)	0.0032 (15)	−0.0072 (16)	−0.0138 (17)
C48	0.0364 (19)	0.041 (2)	0.0356 (17)	0.0037 (14)	−0.0057 (14)	−0.0091 (15)
C49	0.0359 (19)	0.0330 (18)	0.0299 (16)	0.0059 (13)	−0.0057 (13)	−0.0030 (14)
C50	0.0372 (18)	0.0293 (17)	0.0272 (14)	0.0003 (13)	−0.0047 (12)	−0.0028 (14)
C51	0.0356 (19)	0.042 (2)	0.0359 (16)	−0.0007 (15)	−0.0066 (14)	−0.0008 (16)
C52	0.0357 (18)	0.0346 (19)	0.0308 (15)	−0.0002 (14)	0.0008 (13)	−0.0012 (14)
C53	0.0374 (19)	0.0313 (18)	0.0350 (16)	0.0018 (14)	0.0043 (13)	−0.0020 (14)
C54	0.0384 (19)	0.0302 (17)	0.0278 (15)	−0.0014 (14)	0.0007 (13)	−0.0018 (14)
C55	0.040 (2)	0.038 (2)	0.0265 (15)	−0.0040 (14)	−0.0009 (13)	0.0002 (15)
C56	0.0339 (18)	0.0365 (19)	0.0355 (16)	−0.0007 (14)	−0.0024 (13)	−0.0016 (15)
C57	0.0348 (19)	0.037 (2)	0.0393 (17)	−0.0021 (14)	−0.0036 (14)	−0.0034 (16)
C58	0.0351 (19)	0.039 (2)	0.0394 (18)	−0.0033 (14)	0.0067 (14)	−0.0044 (16)
C59	0.0336 (19)	0.045 (2)	0.0343 (17)	−0.0015 (15)	0.0044 (14)	−0.0063 (15)
C60	0.045 (2)	0.054 (2)	0.0405 (19)	0.0015 (18)	0.0050 (16)	−0.0073 (19)
C61	0.0361 (19)	0.051 (2)	0.0267 (16)	0.0068 (15)	−0.0050 (13)	−0.0062 (16)
C62	0.0342 (19)	0.053 (2)	0.0287 (16)	0.0076 (15)	0.0045 (13)	0.0057 (16)
C63	0.045 (2)	0.035 (2)	0.0305 (16)	0.0010 (15)	0.0038 (14)	0.0025 (15)
C64	0.0330 (17)	0.0323 (17)	0.0273 (14)	0.0005 (13)	−0.0005 (12)	−0.0018 (14)
C65	0.0363 (19)	0.036 (2)	0.0385 (17)	−0.0037 (14)	0.0051 (14)	−0.0007 (15)
C66	0.043 (2)	0.0360 (19)	0.0363 (17)	−0.0049 (15)	0.0045 (14)	−0.0037 (16)
C67	0.039 (2)	0.048 (2)	0.0373 (18)	0.0025 (16)	−0.0026 (15)	0.0055 (17)
C68	0.054 (2)	0.049 (2)	0.0353 (18)	−0.0055 (18)	0.0058 (16)	0.0086 (17)
C91	0.063 (3)	0.074 (3)	0.054 (2)	0.010 (2)	−0.009 (2)	0.006 (2)
C92	0.055 (3)	0.070 (3)	0.040 (2)	−0.011 (2)	0.0056 (17)	−0.009 (2)
C93	0.062 (3)	0.059 (3)	0.047 (2)	0.005 (2)	0.0027 (19)	−0.006 (2)
O29	0.0408 (14)	0.0380 (14)	0.0399 (12)	−0.0040 (10)	0.0018 (10)	−0.0058 (11)
O30	0.0398 (14)	0.0420 (15)	0.0248 (10)	−0.0011 (10)	−0.0013 (9)	−0.0061 (10)

O31	0.0458 (15)	0.0481 (16)	0.0304 (11)	0.0003 (11)	0.0020 (10)	−0.0063 (11)
O32	0.0484 (16)	0.0475 (17)	0.0430 (13)	0.0119 (12)	−0.0078 (11)	−0.0213 (13)
O33	0.0441 (14)	0.0381 (14)	0.0248 (10)	−0.0002 (10)	−0.0054 (9)	−0.0070 (10)
O34	0.0400 (13)	0.0299 (12)	0.0262 (10)	0.0038 (9)	−0.0032 (9)	−0.0010 (10)
O35	0.0431 (14)	0.0442 (15)	0.0240 (11)	−0.0009 (10)	−0.0016 (9)	−0.0012 (10)
O36	0.0469 (16)	0.0340 (15)	0.0674 (17)	−0.0005 (12)	−0.0067 (13)	0.0014 (13)
O37	0.087 (2)	0.061 (2)	0.0342 (14)	0.0004 (16)	−0.0051 (14)	−0.0127 (14)
O69	0.0395 (13)	0.0356 (13)	0.0288 (11)	−0.0005 (10)	−0.0046 (9)	−0.0038 (10)
O70	0.0380 (14)	0.0465 (15)	0.0335 (11)	−0.0037 (11)	−0.0004 (10)	−0.0115 (11)
O71	0.0367 (14)	0.0381 (14)	0.0392 (12)	0.0026 (10)	−0.0037 (10)	−0.0061 (11)
O72	0.0411 (15)	0.0566 (18)	0.0393 (13)	0.0034 (12)	−0.0022 (11)	−0.0137 (13)
O73	0.0387 (13)	0.0363 (13)	0.0268 (10)	0.0000 (10)	−0.0040 (9)	−0.0010 (10)
O74	0.0380 (13)	0.0364 (13)	0.0279 (11)	−0.0013 (10)	0.0026 (9)	−0.0001 (10)
O75	0.0453 (15)	0.0426 (15)	0.0372 (12)	−0.0043 (11)	0.0011 (10)	−0.0097 (12)
O76	0.0480 (16)	0.0650 (19)	0.0347 (12)	0.0074 (13)	−0.0004 (11)	0.0117 (13)
O77	0.0543 (17)	0.0341 (15)	0.0559 (15)	0.0035 (12)	0.0029 (12)	−0.0133 (13)
O94	0.066 (2)	0.153 (4)	0.0485 (17)	−0.010 (2)	0.0044 (15)	−0.023 (2)
O95	0.100 (13)	0.038 (8)	0.054 (8)	−0.014 (8)	0.031 (8)	−0.001 (6)

Geometric parameters (Å, º)

C1—O29	1.231 (4)	C44—C45	1.515 (6)
C1—C2	1.470 (5)	C44—H44A	0.9900
C1—C10	1.535 (5)	C44—H44B	0.9900
C2—C3	1.333 (5)	C45—C46	1.335 (6)
C2—H2	0.9500	C45—C50	1.530 (5)
C3—C4	1.493 (6)	C46—C47	1.478 (6)
C3—H3	0.9500	C46—H46	0.9500
C4—C5	1.524 (5)	C47—C48	1.530 (5)
C4—H4A	0.9900	C47—H47A	0.9900
C4—H4B	0.9900	C47—H47B	0.9900
C5—C6	1.313 (6)	C48—C61	1.532 (6)
C5—C10	1.528 (5)	C48—C49	1.552 (5)
C6—C7	1.490 (6)	C48—H48	1.0000
C6—H6	0.9500	C49—C50	1.537 (5)
C7—C8	1.538 (5)	C49—C52	1.548 (5)
C7—H7A	0.9900	C49—H49	1.0000
C7—H7B	0.9900	C50—C51	1.562 (5)
C8—C9	1.530 (5)	C51—H51A	0.9800
C8—C21	1.541 (5)	C51—H51B	0.9800
C8—H8	1.0000	C51—H51C	0.9800
C9—C12	1.558 (4)	C52—C53	1.558 (5)
C9—C10	1.562 (5)	C52—H52A	0.9900
C9—H9	1.0000	C52—H52B	0.9900
C10—C11	1.549 (5)	C53—C54	1.520 (5)
C11—H11A	0.9800	C53—H53A	0.9900
C11—H11B	0.9800	C53—H53B	0.9900
C11—H11C	0.9800	C54—O74	1.424 (4)
C12—C13	1.558 (5)	C54—C64	1.539 (5)
C12—H12A	0.9900	C54—C55	1.545 (5)

C12—H12B	0.9900	C55—O75	1.194 (4)
C13—C14	1.515 (5)	C55—O70	1.352 (5)
C13—H13A	0.9900	C56—O70	1.467 (4)
C13—H13B	0.9900	C56—C67	1.509 (5)
C14—O34	1.417 (4)	C56—C57	1.540 (5)
C14—C24	1.537 (5)	C56—C64	1.563 (4)
C14—C15	1.553 (4)	C57—O71	1.440 (5)
C15—O35	1.211 (4)	C57—C66	1.496 (5)
C15—O30	1.334 (4)	C57—H57	1.0000
C16—O30	1.473 (4)	C58—O77	1.199 (5)
C16—C27	1.518 (5)	C58—O71	1.352 (4)
C16—C17	1.533 (5)	C58—C59	1.511 (5)
C16—C24	1.545 (5)	C59—C60	1.522 (5)
C17—O31	1.459 (4)	C59—C65	1.557 (5)
C17—C26	1.501 (6)	C59—H59	1.0000
C17—H17	1.0000	C60—O72	1.457 (5)
C18—O37	1.212 (5)	C60—H60A	0.9900
C18—O31	1.341 (5)	C60—H60B	0.9900
C18—C19	1.506 (6)	C61—O72	1.405 (4)
C19—C20	1.547 (5)	C61—O73	1.447 (4)
C19—C25	1.569 (5)	C61—C62	1.527 (6)
C19—H19	1.0000	C62—O76	1.216 (4)
C20—O32	1.440 (5)	C62—C63	1.507 (5)
C20—H20A	0.9900	C63—C65	1.539 (5)
C20—H20B	0.9900	C63—C64	1.553 (5)
C21—O32	1.409 (4)	C63—H63	1.0000
C21—O33	1.433 (5)	C64—O73	1.420 (4)
C21—C22	1.534 (5)	C65—C68	1.525 (5)
C22—O36	1.206 (5)	C65—C66	1.531 (5)
C22—C23	1.509 (5)	C66—H66A	0.9900
C23—C24	1.541 (5)	C66—H66B	0.9900
C23—C25	1.552 (5)	C67—H67A	0.9800
C23—H23	1.0000	C67—H67B	0.9800
C24—O33	1.436 (4)	C67—H67C	0.9800
C25—C28	1.517 (6)	C68—H68A	0.9800
C25—C26	1.524 (5)	C68—H68B	0.9800
C26—H26A	0.9900	C68—H68C	0.9800
C26—H26B	0.9900	C91—C92	1.492 (7)
C27—H27A	0.9800	C91—H91A	0.9800
C27—H27B	0.9800	C91—H91B	0.9800
C27—H27C	0.9800	C91—H91C	0.9800
C28—H28A	0.9800	C92—O94	1.231 (5)
C28—H28B	0.9800	C92—C93	1.469 (6)
C28—H28C	0.9800	C93—H93A	0.9800
C41—O69	1.218 (4)	C93—H93B	0.9800
C41—C42	1.464 (5)	C93—H93C	0.9800
C41—C50	1.549 (5)	O34—H34	0.8400
C42—C43	1.335 (6)	O74—H74	0.8400
C42—H42	0.9500	O95—H95A	0.8400

C43—C44	1.468 (6)	O95—H95B	0.86 (14)
C43—H43	0.9500		
O29—C1—C2	120.0 (4)	C45—C44—H44B	108.6
O29—C1—C10	121.9 (3)	H44A—C44—H44B	107.6
C2—C1—C10	118.1 (3)	C46—C45—C44	123.3 (4)
C3—C2—C1	120.8 (4)	C46—C45—C50	121.3 (3)
C3—C2—H2	119.6	C44—C45—C50	115.4 (3)
C1—C2—H2	119.6	C45—C46—C47	125.2 (4)
C2—C3—C4	123.6 (4)	C45—C46—H46	117.4
C2—C3—H3	118.2	C47—C46—H46	117.4
C4—C3—H3	118.2	C46—C47—C48	112.7 (3)
C3—C4—C5	113.2 (3)	C46—C47—H47A	109.0
C3—C4—H4A	108.9	C48—C47—H47A	109.0
C5—C4—H4A	108.9	C46—C47—H47B	109.0
C3—C4—H4B	108.9	C48—C47—H47B	109.0
C5—C4—H4B	108.9	H47A—C47—H47B	107.8
H4A—C4—H4B	107.7	C47—C48—C61	108.7 (3)
C6—C5—C4	121.9 (3)	C47—C48—C49	109.0 (3)
C6—C5—C10	122.6 (3)	C61—C48—C49	114.2 (3)
C4—C5—C10	115.5 (3)	C47—C48—H48	108.2
C5—C6—C7	124.5 (3)	C61—C48—H48	108.2
C5—C6—H6	117.7	C49—C48—H48	108.2
C7—C6—H6	117.7	C50—C49—C52	110.4 (3)
C6—C7—C8	111.8 (3)	C50—C49—C48	111.3 (3)
C6—C7—H7A	109.3	C52—C49—C48	113.8 (3)
C8—C7—H7A	109.3	C50—C49—H49	107.0
C6—C7—H7B	109.3	C52—C49—H49	107.0
C8—C7—H7B	109.3	C48—C49—H49	107.0
H7A—C7—H7B	107.9	C45—C50—C49	112.9 (3)
C9—C8—C7	109.5 (3)	C45—C50—C41	106.7 (3)
C9—C8—C21	114.3 (3)	C49—C50—C41	108.7 (3)
C7—C8—C21	107.7 (3)	C45—C50—C51	106.9 (3)
C9—C8—H8	108.4	C49—C50—C51	113.5 (3)
C7—C8—H8	108.4	C41—C50—C51	107.9 (3)
C21—C8—H8	108.4	C50—C51—H51A	109.5
C8—C9—C12	115.9 (3)	C50—C51—H51B	109.5
C8—C9—C10	110.7 (3)	H51A—C51—H51B	109.5
C12—C9—C10	109.5 (3)	C50—C51—H51C	109.5
C8—C9—H9	106.7	H51A—C51—H51C	109.5
C12—C9—H9	106.7	H51B—C51—H51C	109.5
C10—C9—H9	106.7	C49—C52—C53	120.6 (3)
C5—C10—C1	106.1 (3)	C49—C52—H52A	107.2
C5—C10—C11	108.0 (3)	C53—C52—H52A	107.2
C1—C10—C11	108.9 (3)	C49—C52—H52B	107.2
C5—C10—C9	112.8 (3)	C53—C52—H52B	107.2
C1—C10—C9	107.7 (3)	H52A—C52—H52B	106.8
C11—C10—C9	113.1 (3)	C54—C53—C52	119.2 (3)
C10—C11—H11A	109.5	C54—C53—H53A	107.5

C10—C11—H11B	109.5	C52—C53—H53A	107.5
H11A—C11—H11B	109.5	C54—C53—H53B	107.5
C10—C11—H11C	109.5	C52—C53—H53B	107.5
H11A—C11—H11C	109.5	H53A—C53—H53B	107.0
H11B—C11—H11C	109.5	O74—C54—C53	111.7 (3)
C9—C12—C13	120.4 (3)	O74—C54—C64	107.5 (3)
C9—C12—H12A	107.2	C53—C54—C64	120.0 (3)
C13—C12—H12A	107.2	O74—C54—C55	106.1 (2)
C9—C12—H12B	107.2	C53—C54—C55	110.8 (3)
C13—C12—H12B	107.2	C64—C54—C55	99.3 (3)
H12A—C12—H12B	106.9	O75—C55—O70	122.0 (3)
C14—C13—C12	120.1 (3)	O75—C55—C54	128.6 (3)
C14—C13—H13A	107.3	O70—C55—C54	109.3 (3)
C12—C13—H13A	107.3	O70—C56—C67	109.3 (3)
C14—C13—H13B	107.3	O70—C56—C57	104.8 (3)
C12—C13—H13B	107.3	C67—C56—C57	109.6 (3)
H13A—C13—H13B	106.9	O70—C56—C64	101.4 (3)
O34—C14—C13	111.7 (3)	C67—C56—C64	116.1 (3)
O34—C14—C24	107.5 (3)	C57—C56—C64	114.6 (3)
C13—C14—C24	120.4 (3)	O71—C57—C66	110.7 (3)
O34—C14—C15	105.6 (3)	O71—C57—C56	107.5 (3)
C13—C14—C15	112.2 (3)	C66—C57—C56	115.1 (3)
C24—C14—C15	97.9 (3)	O71—C57—H57	107.7
O35—C15—O30	122.6 (3)	C66—C57—H57	107.7
O35—C15—C14	126.7 (3)	C56—C57—H57	107.7
O30—C15—C14	110.7 (3)	O77—C58—O71	118.1 (3)
O30—C16—C27	109.6 (3)	O77—C58—C59	122.6 (3)
O30—C16—C17	104.7 (2)	O71—C58—C59	119.0 (3)
C27—C16—C17	109.4 (3)	C58—C59—C60	108.8 (3)
O30—C16—C24	100.7 (3)	C58—C59—C65	117.8 (3)
C27—C16—C24	116.0 (3)	C60—C59—C65	118.3 (3)
C17—C16—C24	115.2 (3)	C58—C59—H59	103.1
O31—C17—C26	110.6 (3)	C60—C59—H59	103.1
O31—C17—C16	107.3 (3)	C65—C59—H59	103.1
C26—C17—C16	114.6 (3)	O72—C60—C59	118.3 (3)
O31—C17—H17	108.1	O72—C60—H60A	107.7
C26—C17—H17	108.1	C59—C60—H60A	107.7
C16—C17—H17	108.1	O72—C60—H60B	107.7
O37—C18—O31	117.0 (4)	C59—C60—H60B	107.7
O37—C18—C19	122.1 (4)	H60A—C60—H60B	107.1
O31—C18—C19	120.5 (3)	O72—C61—O73	108.1 (3)
C18—C19—C20	110.3 (3)	O72—C61—C62	107.4 (3)
C18—C19—C25	117.1 (3)	O73—C61—C62	104.9 (3)
C20—C19—C25	117.8 (4)	O72—C61—C48	109.1 (3)
C18—C19—H19	103.0	O73—C61—C48	110.2 (3)
C20—C19—H19	103.0	C62—C61—C48	116.7 (3)
C25—C19—H19	103.0	O76—C62—C63	127.1 (4)
O32—C20—C19	117.6 (3)	O76—C62—C61	125.3 (4)
O32—C20—H20A	107.9	C63—C62—C61	107.1 (3)

C19—C20—H20A	107.9	C62—C63—C65	108.1 (3)
O32—C20—H20B	107.9	C62—C63—C64	100.2 (3)
C19—C20—H20B	107.9	C65—C63—C64	115.2 (3)
H20A—C20—H20B	107.2	C62—C63—H63	110.9
O32—C21—O33	108.2 (3)	C65—C63—H63	110.9
O32—C21—C22	108.8 (3)	C64—C63—H63	110.9
O33—C21—C22	104.8 (3)	O73—C64—C54	115.3 (3)
O32—C21—C8	108.3 (3)	O73—C64—C63	105.2 (2)
O33—C21—C8	110.8 (3)	C54—C64—C63	110.2 (3)
C22—C21—C8	115.7 (3)	O73—C64—C56	110.5 (3)
O36—C22—C23	127.9 (3)	C54—C64—C56	101.9 (2)
O36—C22—C21	125.4 (3)	C63—C64—C56	114.0 (3)
C23—C22—C21	106.4 (3)	C68—C65—C66	109.6 (3)
C22—C23—C24	100.3 (3)	C68—C65—C63	108.6 (3)
C22—C23—C25	109.1 (3)	C66—C65—C63	107.2 (3)
C24—C23—C25	115.3 (3)	C68—C65—C59	110.6 (3)
C22—C23—H23	110.6	C66—C65—C59	106.5 (3)
C24—C23—H23	110.6	C63—C65—C59	114.2 (3)
C25—C23—H23	110.6	C57—C66—C65	109.9 (3)
O33—C24—C14	114.5 (3)	C57—C66—H66A	109.7
O33—C24—C23	104.4 (3)	C65—C66—H66A	109.7
C14—C24—C23	110.4 (3)	C57—C66—H66B	109.7
O33—C24—C16	110.3 (3)	C65—C66—H66B	109.7
C14—C24—C16	103.3 (3)	H66A—C66—H66B	108.2
C23—C24—C16	114.3 (3)	C56—C67—H67A	109.5
C28—C25—C26	110.3 (3)	C56—C67—H67B	109.5
C28—C25—C23	108.4 (3)	H67A—C67—H67B	109.5
C26—C25—C23	106.4 (3)	C56—C67—H67C	109.5
C28—C25—C19	110.2 (3)	H67A—C67—H67C	109.5
C26—C25—C19	107.3 (3)	H67B—C67—H67C	109.5
C23—C25—C19	114.2 (3)	C65—C68—H68A	109.5
C17—C26—C25	110.1 (3)	C65—C68—H68B	109.5
C17—C26—H26A	109.6	H68A—C68—H68B	109.5
C25—C26—H26A	109.6	C65—C68—H68C	109.5
C17—C26—H26B	109.6	H68A—C68—H68C	109.5
C25—C26—H26B	109.6	H68B—C68—H68C	109.5
H26A—C26—H26B	108.1	C92—C91—H91A	109.5
C16—C27—H27A	109.5	C92—C91—H91B	109.5
C16—C27—H27B	109.5	H91A—C91—H91B	109.5
H27A—C27—H27B	109.5	C92—C91—H91C	109.5
C16—C27—H27C	109.5	H91A—C91—H91C	109.5
H27A—C27—H27C	109.5	H91B—C91—H91C	109.5
H27B—C27—H27C	109.5	O94—C92—C93	120.2 (5)
C25—C28—H28A	109.5	O94—C92—C91	122.0 (4)
C25—C28—H28B	109.5	C93—C92—C91	117.8 (4)
H28A—C28—H28B	109.5	C92—C93—H93A	109.5
C25—C28—H28C	109.5	C92—C93—H93B	109.5
H28A—C28—H28C	109.5	H93A—C93—H93B	109.5
H28B—C28—H28C	109.5	C92—C93—H93C	109.5

O69—C41—C42	121.9 (3)	H93A—C93—H93C	109.5
O69—C41—C50	120.6 (3)	H93B—C93—H93C	109.5
C42—C41—C50	117.5 (3)	C15—O30—C16	111.3 (3)
C43—C42—C41	121.3 (4)	C18—O31—C17	120.8 (3)
C43—C42—H42	119.4	C21—O32—C20	118.2 (3)
C41—C42—H42	119.4	C21—O33—C24	110.3 (3)
C42—C43—C44	123.5 (4)	C14—O34—H34	109.5
C42—C43—H43	118.2	C55—O70—C56	111.8 (3)
C44—C43—H43	118.2	C58—O71—C57	120.5 (3)
C43—C44—C45	114.5 (3)	C61—O72—C60	118.1 (3)
C43—C44—H44A	108.6	C64—O73—C61	110.1 (3)
C45—C44—H44A	108.6	C54—O74—H74	109.5
C43—C44—H44B	108.6	H95A—O95—H95B	110.1

O29—C1—C2—C3	156.9 (4)	C46—C45—C50—C51	−115.3 (4)
C10—C1—C2—C3	−21.1 (6)	C44—C45—C50—C51	63.4 (4)
C1—C2—C3—C4	2.5 (7)	C52—C49—C50—C45	−169.0 (3)
C2—C3—C4—C5	−11.2 (6)	C48—C49—C50—C45	−41.6 (4)
C3—C4—C5—C6	−140.3 (4)	C52—C49—C50—C41	72.9 (3)
C3—C4—C5—C10	38.9 (5)	C48—C49—C50—C41	−159.8 (3)
C4—C5—C6—C7	−178.6 (4)	C52—C49—C50—C51	−47.1 (4)
C10—C5—C6—C7	2.3 (6)	C48—C49—C50—C51	80.2 (4)
C5—C6—C7—C8	19.2 (6)	O69—C41—C50—C45	−136.7 (3)
C6—C7—C8—C9	−50.0 (4)	C42—C41—C50—C45	43.0 (4)
C6—C7—C8—C21	−174.9 (3)	O69—C41—C50—C49	−14.8 (4)
C7—C8—C9—C12	−174.0 (3)	C42—C41—C50—C49	165.0 (3)
C21—C8—C9—C12	−53.1 (4)	O69—C41—C50—C51	108.7 (3)
C7—C8—C9—C10	60.5 (4)	C42—C41—C50—C51	−71.6 (4)
C21—C8—C9—C10	−178.5 (3)	C50—C49—C52—C53	−136.6 (3)
C6—C5—C10—C1	125.6 (4)	C48—C49—C52—C53	97.4 (4)
C4—C5—C10—C1	−53.6 (4)	C49—C52—C53—C54	−18.1 (5)
C6—C5—C10—C11	−117.7 (4)	C52—C53—C54—O74	66.3 (4)
C4—C5—C10—C11	63.1 (4)	C52—C53—C54—C64	−60.8 (4)
C6—C5—C10—C9	8.0 (5)	C52—C53—C54—C55	−175.6 (3)
C4—C5—C10—C9	−171.2 (3)	O74—C54—C55—O75	91.8 (4)
O29—C1—C10—C5	−133.3 (3)	C53—C54—C55—O75	−29.6 (5)
C2—C1—C10—C5	44.6 (4)	C64—C54—C55—O75	−156.8 (4)
O29—C1—C10—C11	110.7 (4)	O74—C54—C55—O70	−84.3 (3)
C2—C1—C10—C11	−71.4 (4)	C53—C54—C55—O70	154.3 (3)
O29—C1—C10—C9	−12.3 (4)	C64—C54—C55—O70	27.1 (3)
C2—C1—C10—C9	165.6 (3)	O70—C56—C57—O71	−166.2 (2)
C8—C9—C10—C5	−39.2 (4)	C67—C56—C57—O71	−49.0 (3)
C12—C9—C10—C5	−168.3 (3)	C64—C56—C57—O71	83.5 (3)
C8—C9—C10—C1	−156.0 (3)	O70—C56—C57—C66	69.9 (3)
C12—C9—C10—C1	75.0 (3)	C67—C56—C57—C66	−172.9 (3)
C8—C9—C10—C11	83.6 (4)	C64—C56—C57—C66	−40.4 (4)
C12—C9—C10—C11	−45.4 (4)	O77—C58—C59—C60	−31.1 (5)
C8—C9—C12—C13	92.6 (4)	O71—C58—C59—C60	155.6 (3)
C10—C9—C12—C13	−141.3 (3)	O77—C58—C59—C65	−169.3 (3)

C9—C12—C13—C14	−11.1 (5)	O71—C58—C59—C65	17.4 (5)
C12—C13—C14—O34	62.0 (4)	C58—C59—C60—O72	−60.6 (4)
C12—C13—C14—C24	−65.3 (4)	C65—C59—C60—O72	77.4 (5)
C12—C13—C14—C15	−179.6 (3)	C47—C48—C61—O72	−50.7 (4)
O34—C14—C15—O35	91.5 (4)	C49—C48—C61—O72	−172.7 (3)
C13—C14—C15—O35	−30.4 (5)	C47—C48—C61—O73	67.9 (4)
C24—C14—C15—O35	−157.8 (4)	C49—C48—C61—O73	−54.1 (4)
O34—C14—C15—O30	−86.4 (3)	C47—C48—C61—C62	−172.6 (3)
C13—C14—C15—O30	151.7 (3)	C49—C48—C61—C62	65.4 (4)
C24—C14—C15—O30	24.3 (3)	O72—C61—C62—O76	−70.4 (4)
O30—C16—C17—O31	−167.2 (3)	O73—C61—C62—O76	174.8 (3)
C27—C16—C17—O31	−49.8 (4)	C48—C61—C62—O76	52.4 (5)
C24—C16—C17—O31	83.2 (3)	O72—C61—C62—C63	102.5 (3)
O30—C16—C17—C26	69.6 (3)	O73—C61—C62—C63	−12.3 (4)
C27—C16—C17—C26	−172.9 (3)	C48—C61—C62—C63	−134.6 (3)
C24—C16—C17—C26	−40.0 (4)	O76—C62—C63—C65	80.2 (5)
O37—C18—C19—C20	−35.5 (6)	C61—C62—C63—C65	−92.5 (3)
O31—C18—C19—C20	151.8 (4)	O76—C62—C63—C64	−158.9 (4)
O37—C18—C19—C25	−173.8 (4)	C61—C62—C63—C64	28.4 (3)
O31—C18—C19—C25	13.4 (5)	O74—C54—C64—O73	−47.5 (3)
C18—C19—C20—O32	−60.1 (5)	C53—C54—C64—O73	81.6 (4)
C25—C19—C20—O32	78.0 (5)	C55—C54—C64—O73	−157.7 (3)
C9—C8—C21—O32	−172.5 (3)	O74—C54—C64—C63	−166.3 (3)
C7—C8—C21—O32	−50.6 (4)	C53—C54—C64—C63	−37.3 (4)
C9—C8—C21—O33	−54.1 (4)	C55—C54—C64—C63	83.4 (3)
C7—C8—C21—O33	67.9 (4)	O74—C54—C64—C56	72.3 (3)
C9—C8—C21—C22	65.1 (4)	C53—C54—C64—C56	−158.6 (3)
C7—C8—C21—C22	−173.0 (3)	C55—C54—C64—C56	−38.0 (3)
O32—C21—C22—O36	−72.8 (5)	C62—C63—C64—O73	−35.2 (3)
O33—C21—C22—O36	171.7 (4)	C65—C63—C64—O73	80.5 (3)
C8—C21—C22—O36	49.3 (5)	C62—C63—C64—C54	89.6 (3)
O32—C21—C22—C23	101.5 (3)	C65—C63—C64—C54	−154.7 (3)
O33—C21—C22—C23	−14.0 (4)	C62—C63—C64—C56	−156.5 (3)
C8—C21—C22—C23	−136.4 (3)	C65—C63—C64—C56	−40.8 (4)
O36—C22—C23—C24	−155.3 (4)	O70—C56—C64—O73	160.0 (2)
C21—C22—C23—C24	30.6 (4)	C67—C56—C64—O73	41.7 (4)
O36—C22—C23—C25	83.2 (5)	C57—C56—C64—O73	−87.7 (4)
C21—C22—C23—C25	−90.9 (3)	O70—C56—C64—C54	37.0 (3)
O34—C14—C24—O33	−47.9 (4)	C67—C56—C64—C54	−81.4 (4)
C13—C14—C24—O33	81.4 (4)	C57—C56—C64—C54	149.3 (3)
C15—C14—C24—O33	−157.0 (3)	O70—C56—C64—C63	−81.8 (3)
O34—C14—C24—C23	−165.3 (3)	C67—C56—C64—C63	159.9 (3)
C13—C14—C24—C23	−36.1 (4)	C57—C56—C64—C63	30.5 (4)
C15—C14—C24—C23	85.5 (3)	C62—C63—C65—C68	−73.5 (4)
O34—C14—C24—C16	72.1 (3)	C64—C63—C65—C68	175.4 (3)
C13—C14—C24—C16	−158.7 (3)	C62—C63—C65—C66	168.2 (3)
C15—C14—C24—C16	−37.1 (3)	C64—C63—C65—C66	57.1 (4)
C22—C23—C24—O33	−36.8 (3)	C62—C63—C65—C59	50.5 (4)
C25—C23—C24—O33	80.2 (3)	C64—C63—C65—C59	−60.6 (4)

C22—C23—C24—C14	86.7 (3)	C58—C59—C65—C68	−154.5 (3)
C25—C23—C24—C14	−156.3 (3)	C60—C59—C65—C68	71.2 (4)
C22—C23—C24—C16	−157.3 (3)	C58—C59—C65—C66	−35.5 (4)
C25—C23—C24—C16	−40.3 (4)	C60—C59—C65—C66	−169.8 (3)
O30—C16—C24—O33	160.9 (3)	C58—C59—C65—C63	82.6 (4)
C27—C16—C24—O33	42.7 (4)	C60—C59—C65—C63	−51.7 (4)
C17—C16—C24—O33	−87.1 (3)	O71—C57—C66—C65	−63.8 (4)
O30—C16—C24—C14	38.1 (3)	C56—C57—C66—C65	58.4 (4)
C27—C16—C24—C14	−80.0 (3)	C68—C65—C66—C57	177.7 (3)
C17—C16—C24—C14	150.2 (3)	C63—C65—C66—C57	−64.6 (4)
O30—C16—C24—C23	−81.8 (3)	C59—C65—C66—C57	58.0 (4)
C27—C16—C24—C23	160.0 (3)	O35—C15—O30—C16	−178.5 (3)
C17—C16—C24—C23	30.2 (4)	C14—C15—O30—C16	−0.5 (4)
C22—C23—C25—C28	−72.6 (4)	C27—C16—O30—C15	99.2 (3)
C24—C23—C25—C28	175.5 (3)	C17—C16—O30—C15	−143.5 (3)
C22—C23—C25—C26	168.7 (3)	C24—C16—O30—C15	−23.6 (3)
C24—C23—C25—C26	56.8 (4)	O37—C18—O31—C17	170.2 (4)
C22—C23—C25—C19	50.6 (4)	C19—C18—O31—C17	−16.7 (5)
C24—C23—C25—C19	−61.3 (4)	C26—C17—O31—C18	41.0 (4)
C18—C19—C25—C28	−153.8 (3)	C16—C17—O31—C18	−84.6 (4)
C20—C19—C25—C28	71.0 (4)	O33—C21—O32—C20	83.1 (4)
C18—C19—C25—C26	−33.6 (4)	C22—C21—O32—C20	−30.2 (5)
C20—C19—C25—C26	−168.9 (3)	C8—C21—O32—C20	−156.8 (3)
C18—C19—C25—C23	84.0 (4)	C19—C20—O32—C21	−48.2 (5)
C20—C19—C25—C23	−51.3 (5)	O32—C21—O33—C24	−126.4 (3)
O31—C17—C26—C25	−62.8 (4)	C22—C21—O33—C24	−10.5 (4)
C16—C17—C26—C25	58.6 (4)	C8—C21—O33—C24	115.1 (3)
C28—C25—C26—C17	177.9 (3)	C14—C24—O33—C21	−90.4 (3)
C23—C25—C26—C17	−64.7 (4)	C23—C24—O33—C21	30.4 (4)
C19—C25—C26—C17	57.9 (4)	C16—C24—O33—C21	153.6 (3)
O69—C41—C42—C43	160.0 (4)	O75—C55—O70—C56	179.9 (3)
C50—C41—C42—C43	−19.7 (5)	C54—C55—O70—C56	−3.7 (4)
C41—C42—C43—C44	1.5 (6)	C67—C56—O70—C55	102.0 (3)
C42—C43—C44—C45	−10.2 (6)	C57—C56—O70—C55	−140.7 (3)
C43—C44—C45—C46	−143.8 (4)	C64—C56—O70—C55	−21.1 (3)
C43—C44—C45—C50	37.5 (5)	O77—C58—O71—C57	165.4 (3)
C44—C45—C46—C47	−175.7 (3)	C59—C58—O71—C57	−21.0 (5)
C50—C45—C46—C47	2.9 (6)	C66—C57—O71—C58	44.2 (4)
C45—C46—C47—C48	16.7 (5)	C56—C57—O71—C58	−82.4 (4)
C46—C47—C48—C61	−172.2 (3)	O73—C61—O72—C60	82.0 (4)
C46—C47—C48—C49	−47.1 (4)	C62—C61—O72—C60	−30.6 (4)
C47—C48—C49—C50	60.5 (3)	C48—C61—O72—C60	−158.1 (3)
C61—C48—C49—C50	−177.7 (3)	C59—C60—O72—C61	−46.8 (5)
C47—C48—C49—C52	−174.0 (3)	C54—C64—O73—C61	−91.8 (3)
C61—C48—C49—C52	−52.2 (4)	C63—C64—O73—C61	29.8 (3)
C46—C45—C50—C49	10.2 (4)	C56—C64—O73—C61	153.3 (3)
C44—C45—C50—C49	−171.1 (3)	O72—C61—O73—C64	−125.7 (3)
C46—C45—C50—C41	129.5 (3)	C62—C61—O73—C64	−11.3 (4)
C44—C45—C50—C41	−51.8 (4)	C48—C61—O73—C64	115.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O34—H34···O69	0.84	1.90	2.735 (3)	171
O74—H74···O29	0.84	2.02	2.806 (3)	156
O95—H95*A*···O37	0.84	1.71	2.513 (2)	161
O95—H95*B*···O74^{i}	0.86 (14)	2.12 (9)	2.924 (3)	155
C52—H52*A*···O76	0.99	2.40	3.232 (4)	141
C91—H91*B*···O36	0.98	2.59	3.456 (6)	147
C17—H17···O75^{ii}	1.00	2.59	3.421 (4)	141
C53—H53*B*···O77^{iii}	0.99	2.45	3.415 (5)	163
C59—H59···O94^{iv}	1.00	2.58	3.463 (5)	147
C63—H63···O77^{iii}	1.00	2.36	3.345 (5)	169

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+1; (ii) *x*+1, *y*, *z*; (iii) −*x*+1, *y*−1/2, −*z*+2; (iv) −*x*+1, *y*+1/2, −*z*+2.

(Y) Physalin B epoxyphysalin B acetone monosolvate

Crystal data

$C_{28}H_{30}O_{9.24}·C_{28}H_{30}O_{9.43}·C_3H_6O$
M_r = 1089.81
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 12.4859 (4) Å
b = 14.1716 (4) Å
c = 14.6559 (7) Å
β = 96.829 (3)°
V = 2574.89 (16) Å^3
Z = 2
F(000) = 1154.7
D_x = 1.406 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 8005 reflections
θ = 3.0–74.2°
μ = 0.88 mm^{-1}
T = 100 K
Plate, colourless
0.45 × 0.24 × 0.01 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3468 pixels mm^{-1}
ω and *φ* scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.699, T_{max} = 1.000
15953 measured reflections
8953 independent reflections
8208 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 66.6°, θ_{min} = 3.0°
h = −10→14
k = −16→16
l = −17→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.175
S = 1.06
8953 reflections
745 parameters
50 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0954P)^2 + 2.1179P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Absolute structure: Flack (1983), 4215 Friedel pairs
Absolute structure parameter: 0.1 (2)

Special details

Experimental. For compound Y, diffraction data were collected on a SuperNova Diffractometer and analyzed with *CrysAlis PRO* software (Agilent, 2012). A dataset of 1136 frames having a width of 1° was collected to a θ-value of 66.60° corresponding to a resolution of 0.844?Å. The crystal belongs to the monoclinic space group P21 with R_{int}-value of 5.41% and a completeness of 99.96%. An empirical absorption correction based on the crystal morphology resulted in Tmin and Tmax of 0.699 and 1.000, respectively.
The structure was solved by direct methods using the *SHELX* program and refined according to the least-quares methods by *SHELXTL* package (Bruker, 2003). Non-hydrogen atoms were located in a fourrier map and refined anisotropically.
#__ type_ start__ end____ width___ exp.time_ 1 omega -69.00 - 3.00 1.0000 3.0000 omega____ theta____ kappa____ phi_____ frames - -40.9779 125.0000 60.0000 66
#__ type_ start__ end____ width___ exp.time_ 2 omega -113.00 - 16.00 1.0000 3.0000 omega____ theta____ kappa____ phi_____ frames - -40.9779 - 38.0000 - 30.0000 97
#__ type_ start__ end____ width___ exp.time_ 3 omega 37.00 72.00 1.0000 3.0000 omega____ theta____ kappa____ phi_____ frames - 40.9779 - 125.0000 - 60.0000 35
#__ type_ start__ end____ width___ exp.time_ 4 omega 89.00 173.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 45.0000 90.0000 84
#__ type_ start__ end____ width___ exp.time_ 5 omega 34.00 77.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 45.0000 - 30.0000 43
#__ type_ start__ end____ width___ exp.time_ 6 omega 38.00 115.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 111.0000 0.0000 77
#__ type_ start__ end____ width___ exp.time_ 7 omega 43.00 77.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 45.0000 150.0000 34
#__ type_ start__ end____ width___ exp.time_ 8 omega 42.00 76.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 45.0000 0.0000 34
#__ type_ start__ end____ width___ exp.time_ 9 omega 100.00 159.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 111.0000 - 86.0000 59
#__ type_ start__ end____ width___ exp.time_ 10 omega 30.00 113.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 105.0000 138.0000 83
#__ type_ start__ end____ width___ exp.time_ 11 omega -93.00 - 4.00 1.0000 3.0000 omega____ theta____ kappa____ phi_____ frames - -41.1029 125.0000 - 180.0000 89
#__ type_ start__ end____ width___ exp.time_ 12 omega -117.00 - 23.00 1.0000 3.0000 omega____ theta____ kappa____ phi_____ frames - -41.1029 - 38.0000 - 120.0000 94
#__ type_ start__ end____ width___ exp.time_ 13 omega 37.00 77.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 45.0000 120.0000 40
#__ type_ start__ end____ width___ exp.time_ 14 omega 33.00 69.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 45.0000 - 120.0000 36
#__ type_ start__ end____ width___ exp.time_ 15 omega 48.00 76.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 45.0000 - 90.0000 28
#__ type_ start__ end____ width___ exp.time_ 16 omega 95.00 166.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 79.0000 26.0000 71
#__ type_ start__ end____ width___ exp.time_ 17 omega 32.00 103.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 - 111.0000 - 93.0000 71
#__ type_ start__ end____ width___ exp.time_ 18 omega 81.00 176.00 1.0000 12.0000 omega____ theta____ kappa____ phi_____ frames - 108.2085 45.0000 - 120.0000 95

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.6702 (3)	0.5285 (3)	0.5734 (3)	0.0297 (8)	
C2	0.5662 (3)	0.5716 (3)	0.5401 (3)	0.0351 (9)	
H2	0.5339	0.6148	0.5782	0.042*	
C3	0.5161 (3)	0.5507 (3)	0.4570 (3)	0.0420 (10)	
H3	0.4501	0.5820	0.4373	0.050*	
C4	0.5570 (3)	0.4822 (3)	0.3940 (3)	0.0410 (10)	
H4AA	0.5538	0.5109	0.3322	0.049*	0.761 (14)
H4AB	0.5093	0.4262	0.3891	0.049*	0.761 (14)
H4BC	0.5561	0.5119	0.3328	0.049*	0.239 (14)
H4BD	0.5074	0.4275	0.3873	0.049*	0.239 (14)
C5A	0.6716 (10)	0.451 (2)	0.4243 (5)	0.0338 (12)	0.761 (14)
C6A	0.7433 (8)	0.4393 (17)	0.3655 (7)	0.0346 (12)	0.761 (14)
H6A	0.7196	0.4500	0.3023	0.042*	0.761 (14)
C5B	0.669 (3)	0.447 (6)	0.4243 (12)	0.0338 (12)	0.239 (14)
C6B	0.745 (2)	0.439 (6)	0.356 (2)	0.0346 (12)	0.239 (14)
H6B	0.7306	0.4728	0.2959	0.042*	0.239 (14)
O39	0.6768 (11)	0.3549 (11)	0.3743 (9)	0.049 (5)	0.239 (14)
C7	0.8588 (3)	0.4111 (3)	0.3903 (3)	0.0380 (10)	
H7AA	0.9051	0.4679	0.3927	0.046*	0.761 (14)
H7AB	0.8810	0.3685	0.3424	0.046*	0.761 (14)
H7BC	0.9038	0.4688	0.3947	0.046*	0.239 (14)
H7BD	0.8862	0.3693	0.3442	0.046*	0.239 (14)
C8	0.8745 (3)	0.3611 (3)	0.4833 (3)	0.0319 (8)	
H8	0.8347	0.2998	0.4773	0.038*	
C9	0.8252 (3)	0.4222 (3)	0.5552 (3)	0.0289 (8)	
H9	0.8596	0.4859	0.5539	0.035*	
C10	0.7021 (3)	0.4369 (3)	0.5271 (3)	0.0311 (8)	
C11	0.6323 (3)	0.3548 (3)	0.5584 (3)	0.0362 (9)	
H11A	0.6549	0.2952	0.5325	0.054*	
H11B	0.5562	0.3665	0.5367	0.054*	
H11C	0.6419	0.3508	0.6256	0.054*	
C12	0.8456 (3)	0.3869 (3)	0.6555 (3)	0.0297 (8)	
H12A	0.8515	0.3173	0.6532	0.036*	
H12B	0.7800	0.4014	0.6848	0.036*	
C13	0.9440 (3)	0.4232 (3)	0.7216 (3)	0.0293 (8)	
H13A	0.9154	0.4637	0.7681	0.035*	
H13B	0.9776	0.3677	0.7545	0.035*	
C14	1.0340 (3)	0.4783 (2)	0.6834 (3)	0.0259 (7)	
C15	1.1242 (3)	0.5061 (2)	0.7597 (3)	0.0275 (8)	
C16	1.2058 (3)	0.4926 (3)	0.6262 (3)	0.0286 (8)	
C17	1.3100 (3)	0.4407 (3)	0.6098 (3)	0.0328 (8)	
H17	1.3725	0.4795	0.6374	0.039*	
C18	1.2641 (4)	0.3678 (3)	0.4594 (3)	0.0392 (10)	
C19	1.2268 (3)	0.2794 (3)	0.5064 (3)	0.0365 (9)	
H19	1.2850	0.2327	0.4981	0.044*	
C20	1.1271 (4)	0.2369 (3)	0.4477 (3)	0.0418 (10)	
H20A	1.1490	0.2190	0.3873	0.050*	

H20B	1.1072	0.1781	0.4780	0.050*	
C21	0.9947 (3)	0.3398 (3)	0.5072 (3)	0.0310 (8)	
C22	1.0264 (3)	0.2804 (3)	0.5932 (3)	0.0303 (8)	
C23	1.1239 (3)	0.3262 (3)	0.6451 (3)	0.0304 (8)	
H23	1.1237	0.3184	0.7129	0.036*	
C24	1.1033 (3)	0.4304 (3)	0.6164 (3)	0.0260 (7)	
C25	1.2271 (4)	0.2821 (3)	0.6129 (3)	0.0359 (9)	
C26	1.3211 (3)	0.3437 (3)	0.6522 (3)	0.0357 (9)	
H26A	1.3222	0.3486	0.7197	0.043*	
H26B	1.3899	0.3147	0.6392	0.043*	
C27	1.1981 (3)	0.5840 (3)	0.5709 (3)	0.0359 (9)	
H27A	1.2695	0.6135	0.5747	0.054*	
H27B	1.1476	0.6271	0.5959	0.054*	
H27C	1.1723	0.5700	0.5065	0.054*	
C28	1.2407 (4)	0.1824 (3)	0.6514 (4)	0.0430 (10)	
H28A	1.1793	0.1436	0.6262	0.065*	
H28B	1.2441	0.1845	0.7185	0.065*	
H28C	1.3076	0.1550	0.6343	0.065*	
C41	0.9755 (3)	0.7097 (2)	0.8370 (3)	0.0282 (8)	
C42	1.0703 (4)	0.7565 (3)	0.8097 (3)	0.0353 (9)	
H42	1.1001	0.7352	0.7566	0.042*	
C43	1.1166 (4)	0.8294 (3)	0.8580 (3)	0.0378 (9)	
H43	1.1776	0.8586	0.8370	0.045*	
C44	1.0771 (4)	0.8664 (3)	0.9423 (3)	0.0435 (11)	
H44A	1.0716	0.9359	0.9375	0.052*	0.570 (13)
H44B	1.1309	0.8514	0.9955	0.052*	0.570 (13)
H44C	1.0659	0.9352	0.9346	0.052*	0.430 (13)
H44D	1.1346	0.8571	0.9941	0.052*	0.430 (13)
C45A	0.969 (3)	0.8271 (17)	0.960 (4)	0.045 (4)	0.570 (13)
C46A	0.893 (2)	0.877 (2)	0.993 (4)	0.041 (3)	0.570 (13)
H46A	0.9094	0.9417	1.0047	0.049*	0.570 (13)
C45B	0.975 (4)	0.824 (2)	0.969 (6)	0.045 (4)	0.430 (13)
C46B	0.894 (3)	0.885 (3)	1.004 (5)	0.041 (3)	0.430 (13)
H46B	0.9004	0.9549	0.9956	0.049*	0.430 (13)
O79	0.9766 (6)	0.8360 (5)	1.0709 (5)	0.040 (3)	0.430 (13)
C47	0.7857 (3)	0.8445 (3)	1.0133 (3)	0.0373 (10)	
H47A	0.7300	0.8646	0.9634	0.045*	0.570 (13)
H47B	0.7688	0.8738	1.0712	0.045*	0.570 (13)
H47C	0.7353	0.8630	0.9590	0.045*	0.430 (13)
H47D	0.7589	0.8727	1.0681	0.045*	0.430 (13)
C48	0.7836 (3)	0.7366 (3)	1.0226 (3)	0.0309 (8)	
H48	0.8306	0.7187	1.0800	0.037*	
C49	0.8314 (3)	0.6921 (2)	0.9399 (3)	0.0265 (8)	
H49	0.7885	0.7168	0.8829	0.032*	
C50	0.9498 (3)	0.7229 (3)	0.9365 (3)	0.0291 (8)	
C51	1.0324 (3)	0.6635 (3)	1.0002 (3)	0.0348 (9)	
H51A	1.0104	0.6616	1.0622	0.052*	
H51B	1.1040	0.6924	1.0026	0.052*	
H51C	1.0348	0.5992	0.9761	0.052*	

C52	0.8225 (3)	0.5833 (3)	0.9364 (3)	0.0263 (7)
H52A	0.8182	0.5610	0.9999	0.032*
H52B	0.8910	0.5587	0.9179	0.032*
C53	0.7285 (3)	0.5356 (3)	0.8734 (3)	0.0262 (7)
H53A	0.7588	0.5114	0.8185	0.031*
H53B	0.7049	0.4801	0.9067	0.031*
C54	0.6282 (3)	0.5926 (3)	0.8397 (2)	0.0264 (7)
C55	0.5420 (3)	0.5302 (3)	0.7855 (3)	0.0279 (8)
C56	0.4490 (3)	0.6507 (3)	0.8497 (3)	0.0296 (8)
C57	0.3507 (3)	0.6438 (3)	0.9029 (3)	0.0326 (8)
H57	0.2860	0.6319	0.8573	0.039*
C58	0.3932 (3)	0.7578 (3)	1.0243 (3)	0.0318 (9)
C59	0.4440 (3)	0.6794 (3)	1.0870 (3)	0.0327 (8)
H59	0.3897	0.6689	1.1310	0.039*
C60	0.5420 (4)	0.7192 (3)	1.1483 (3)	0.0394 (9)
H60A	0.5158	0.7694	1.1870	0.047*
H60B	0.5716	0.6682	1.1900	0.047*
C61	0.6685 (3)	0.7057 (3)	1.0323 (3)	0.0316 (8)
C62	0.6517 (3)	0.6005 (3)	1.0543 (3)	0.0336 (9)
C63	0.5558 (3)	0.5684 (3)	0.9913 (3)	0.0305 (8)
H63	0.5643	0.5013	0.9720	0.037*
C64	0.5592 (3)	0.6362 (3)	0.9094 (3)	0.0267 (7)
C65	0.4551 (3)	0.5800 (3)	1.0433 (3)	0.0315 (8)
C66	0.3563 (3)	0.5663 (3)	0.9728 (3)	0.0324 (8)
H66A	0.3607	0.5045	0.9420	0.039*
H66B	0.2902	0.5668	1.0041	0.039*
C67	0.4398 (3)	0.7395 (3)	0.7900 (3)	0.0358 (9)
H67A	0.3662	0.7447	0.7586	0.054*
H67B	0.4909	0.7355	0.7443	0.054*
H67C	0.4564	0.7951	0.8289	0.054*
C68	0.4577 (4)	0.5033 (3)	1.1166 (3)	0.0385 (9)
H68A	0.5254	0.5075	1.1576	0.058*
H68B	0.4525	0.4412	1.0870	0.058*
H68C	0.3968	0.5120	1.1522	0.058*
C91	0.9182 (5)	0.0191 (5)	0.7537 (4)	0.0598 (14)
H91A	0.9584	0.0026	0.8131	0.090*
H91B	0.9637	0.0581	0.7186	0.090*
H91C	0.8976	−0.0386	0.7192	0.090*
C92	0.8188 (4)	0.0732 (4)	0.7691 (3)	0.0500 (12)
C93	0.7520 (5)	0.1084 (4)	0.6872 (4)	0.0522 (12)
H93A	0.6954	0.1496	0.7058	0.078*
H93B	0.7187	0.0550	0.6519	0.078*
H93C	0.7971	0.1441	0.6490	0.078*
O29	0.7296 (2)	0.5666 (2)	0.63429 (19)	0.0322 (6)
O30	1.2186 (2)	0.51482 (19)	0.72403 (18)	0.0288 (6)
O31	1.3144 (2)	0.4364 (2)	0.51127 (19)	0.0354 (6)
O32	1.0316 (2)	0.2947 (2)	0.4313 (2)	0.0384 (7)
O33	1.0554 (2)	0.42618 (18)	0.52361 (18)	0.0283 (5)
O34	0.9943 (2)	0.56394 (17)	0.64189 (17)	0.0267 (5)

H34	0.9698	0.5980	0.6815	0.040*
O35	1.1169 (2)	0.52281 (19)	0.83868 (17)	0.0298 (6)
O36	0.9844 (2)	0.2071 (2)	0.6105 (2)	0.0407 (7)
O37	1.2584 (3)	0.3742 (3)	0.3777 (2)	0.0512 (8)
O69	0.9184 (2)	0.66049 (18)	0.78285 (18)	0.0288 (6)
O70	0.4432 (2)	0.5673 (2)	0.79051 (19)	0.0326 (6)
O71	0.3366 (2)	0.73502 (19)	0.9444 (2)	0.0326 (6)
O72	0.6302 (2)	0.7580 (2)	1.1036 (2)	0.0383 (7)
O73	0.5991 (2)	0.72288 (18)	0.94852 (18)	0.0276 (5)
O74	0.6522 (2)	0.66562 (19)	0.77836 (17)	0.0273 (5)
H74	0.6896	0.6435	0.7394	0.041*
O75	0.5540 (2)	0.4608 (2)	0.74089 (19)	0.0339 (6)
O76	0.7018 (2)	0.5576 (2)	1.1169 (2)	0.0418 (7)
O77	0.3956 (3)	0.8386 (2)	1.0486 (2)	0.0408 (7)
O94	0.7939 (4)	0.0864 (5)	0.8446 (3)	0.0899 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0266 (19)	0.0288 (19)	0.0333 (19)	−0.0047 (15)	0.0026 (16)	0.0019 (16)
C2	0.029 (2)	0.035 (2)	0.041 (2)	0.0056 (16)	0.0034 (17)	0.0035 (18)
C3	0.026 (2)	0.051 (3)	0.048 (2)	0.0013 (18)	−0.0007 (18)	0.014 (2)
C4	0.037 (2)	0.044 (2)	0.040 (2)	−0.0070 (19)	−0.0059 (18)	0.0053 (19)
C5A	0.037 (2)	0.030 (3)	0.033 (2)	−0.005 (2)	−0.0065 (17)	−0.0039 (16)
C6A	0.043 (2)	0.034 (2)	0.025 (3)	−0.0057 (18)	−0.0062 (19)	−0.002 (3)
C5B	0.037 (2)	0.030 (3)	0.033 (2)	−0.005 (2)	−0.0065 (17)	−0.0039 (16)
C6B	0.043 (2)	0.034 (2)	0.025 (3)	−0.0057 (18)	−0.0062 (19)	−0.002 (3)
O39	0.042 (8)	0.064 (10)	0.043 (8)	−0.005 (7)	0.008 (6)	−0.018 (7)
C7	0.041 (2)	0.039 (2)	0.033 (2)	0.0045 (18)	0.0021 (18)	−0.0096 (18)
C8	0.033 (2)	0.0277 (19)	0.033 (2)	−0.0022 (15)	−0.0032 (16)	−0.0092 (16)
C9	0.0315 (19)	0.0245 (18)	0.0291 (18)	−0.0032 (15)	−0.0035 (15)	−0.0042 (15)
C10	0.032 (2)	0.0233 (18)	0.036 (2)	−0.0036 (15)	−0.0018 (16)	−0.0011 (16)
C11	0.031 (2)	0.031 (2)	0.044 (2)	−0.0068 (16)	−0.0038 (17)	−0.0011 (18)
C12	0.032 (2)	0.0260 (18)	0.0312 (19)	0.0008 (15)	0.0045 (16)	−0.0007 (15)
C13	0.0313 (19)	0.0271 (18)	0.0296 (18)	0.0016 (15)	0.0040 (16)	−0.0015 (15)
C14	0.0297 (18)	0.0198 (17)	0.0263 (18)	−0.0003 (14)	−0.0041 (15)	−0.0021 (14)
C15	0.0310 (19)	0.0190 (17)	0.031 (2)	0.0000 (14)	−0.0029 (15)	0.0022 (14)
C16	0.0309 (19)	0.0275 (18)	0.0269 (18)	0.0000 (15)	0.0016 (15)	−0.0062 (15)
C17	0.031 (2)	0.034 (2)	0.032 (2)	−0.0055 (16)	0.0008 (16)	−0.0084 (16)
C18	0.042 (2)	0.040 (2)	0.036 (2)	0.0088 (19)	0.0014 (18)	−0.0120 (18)
C19	0.032 (2)	0.031 (2)	0.046 (2)	0.0068 (16)	0.0033 (18)	−0.0128 (18)
C20	0.040 (2)	0.036 (2)	0.048 (3)	0.0054 (18)	−0.0001 (19)	−0.0167 (19)
C21	0.033 (2)	0.0249 (18)	0.034 (2)	0.0010 (15)	0.0002 (16)	−0.0092 (16)
C22	0.0284 (19)	0.024 (2)	0.039 (2)	0.0048 (15)	0.0062 (16)	−0.0063 (16)
C23	0.035 (2)	0.0216 (18)	0.033 (2)	0.0004 (15)	−0.0042 (16)	−0.0005 (15)
C24	0.0256 (18)	0.0241 (18)	0.0268 (17)	−0.0003 (14)	−0.0026 (14)	−0.0020 (14)
C25	0.036 (2)	0.025 (2)	0.045 (2)	0.0030 (16)	−0.0025 (18)	−0.0035 (17)
C26	0.029 (2)	0.035 (2)	0.041 (2)	0.0064 (17)	−0.0031 (17)	−0.0059 (18)
C27	0.036 (2)	0.032 (2)	0.039 (2)	−0.0058 (17)	0.0057 (17)	0.0016 (18)
C28	0.037 (2)	0.033 (2)	0.056 (3)	0.0047 (18)	−0.006 (2)	−0.006 (2)

C41	0.0312 (19)	0.0171 (17)	0.0338 (19)	0.0042 (14)	−0.0064 (15)	0.0017 (15)
C42	0.039 (2)	0.031 (2)	0.035 (2)	−0.0015 (17)	0.0006 (17)	0.0030 (17)
C43	0.038 (2)	0.029 (2)	0.045 (2)	−0.0072 (17)	0.0009 (18)	0.0070 (18)
C44	0.048 (2)	0.0209 (19)	0.058 (3)	−0.0053 (17)	−0.007 (2)	−0.0060 (18)
C45A	0.048 (4)	0.029 (3)	0.057 (10)	−0.006 (3)	−0.002 (6)	−0.014 (4)
C46A	0.046 (2)	0.020 (4)	0.054 (11)	0.001 (2)	−0.005 (3)	−0.018 (4)
C45B	0.048 (4)	0.029 (3)	0.057 (10)	−0.006 (3)	−0.002 (6)	−0.014 (4)
C46B	0.046 (2)	0.020 (4)	0.054 (11)	0.001 (2)	−0.005 (3)	−0.018 (4)
O79	0.041 (4)	0.042 (4)	0.034 (4)	−0.005 (3)	−0.003 (3)	−0.008 (3)
C47	0.034 (2)	0.028 (2)	0.047 (2)	0.0066 (16)	−0.0072 (18)	−0.0164 (18)
C48	0.0312 (19)	0.0292 (19)	0.0301 (19)	0.0045 (15)	−0.0056 (16)	−0.0066 (15)
C49	0.0311 (19)	0.0199 (17)	0.0265 (17)	0.0012 (14)	−0.0054 (14)	−0.0012 (13)
C50	0.033 (2)	0.0200 (17)	0.0330 (19)	0.0008 (15)	−0.0026 (15)	−0.0059 (15)
C51	0.031 (2)	0.039 (2)	0.0320 (19)	−0.0002 (17)	−0.0050 (16)	0.0022 (17)
C52	0.0272 (18)	0.0218 (17)	0.0287 (18)	0.0010 (14)	−0.0018 (14)	0.0000 (14)
C53	0.0259 (18)	0.0214 (17)	0.0316 (18)	−0.0018 (14)	0.0044 (15)	−0.0038 (14)
C54	0.0303 (19)	0.0229 (17)	0.0251 (17)	−0.0024 (15)	−0.0001 (14)	−0.0011 (14)
C55	0.0326 (19)	0.0231 (18)	0.0274 (17)	−0.0056 (15)	0.0010 (15)	0.0009 (15)
C56	0.0280 (18)	0.030 (2)	0.0291 (18)	−0.0002 (15)	−0.0032 (15)	−0.0038 (16)
C57	0.031 (2)	0.0265 (19)	0.038 (2)	−0.0012 (15)	−0.0024 (16)	−0.0076 (17)
C58	0.0240 (18)	0.031 (2)	0.041 (2)	−0.0031 (15)	0.0075 (16)	−0.0050 (17)
C59	0.033 (2)	0.0290 (19)	0.036 (2)	−0.0006 (16)	0.0044 (17)	−0.0037 (16)
C60	0.044 (2)	0.039 (2)	0.036 (2)	−0.0005 (19)	0.0059 (18)	−0.0074 (19)
C61	0.035 (2)	0.035 (2)	0.0238 (18)	0.0029 (16)	−0.0035 (15)	−0.0064 (16)
C62	0.031 (2)	0.041 (2)	0.0284 (19)	0.0027 (17)	0.0020 (16)	0.0025 (17)
C63	0.034 (2)	0.0264 (18)	0.0297 (19)	0.0019 (16)	0.0000 (16)	0.0029 (15)
C64	0.0322 (19)	0.0218 (16)	0.0254 (17)	−0.0006 (14)	0.0005 (15)	−0.0008 (14)
C65	0.035 (2)	0.0269 (19)	0.0334 (19)	−0.0003 (16)	0.0065 (16)	0.0003 (16)
C66	0.033 (2)	0.0258 (19)	0.040 (2)	−0.0077 (16)	0.0107 (17)	−0.0091 (17)
C67	0.032 (2)	0.039 (2)	0.035 (2)	0.0016 (17)	−0.0045 (17)	0.0042 (17)
C68	0.044 (2)	0.035 (2)	0.038 (2)	−0.0048 (18)	0.0122 (18)	0.0024 (18)
C91	0.060 (3)	0.061 (3)	0.057 (3)	0.003 (3)	0.001 (3)	0.007 (3)
C92	0.047 (3)	0.064 (3)	0.039 (2)	−0.022 (2)	0.006 (2)	−0.006 (2)
C93	0.056 (3)	0.050 (3)	0.052 (3)	0.003 (2)	0.008 (2)	−0.007 (2)
O29	0.0300 (14)	0.0302 (13)	0.0365 (14)	−0.0014 (11)	0.0042 (11)	−0.0061 (12)
O30	0.0280 (13)	0.0277 (13)	0.0299 (13)	−0.0026 (11)	0.0005 (11)	−0.0053 (11)
O31	0.0353 (15)	0.0380 (16)	0.0329 (14)	−0.0012 (12)	0.0043 (12)	−0.0069 (12)
O32	0.0424 (16)	0.0335 (15)	0.0380 (16)	0.0027 (12)	−0.0008 (13)	−0.0134 (13)
O33	0.0323 (14)	0.0248 (13)	0.0267 (12)	−0.0015 (11)	−0.0010 (11)	−0.0036 (10)
O34	0.0331 (14)	0.0205 (12)	0.0255 (12)	0.0026 (10)	−0.0010 (10)	−0.0017 (10)
O35	0.0358 (14)	0.0286 (13)	0.0234 (13)	−0.0017 (11)	−0.0024 (10)	−0.0013 (11)
O36	0.0390 (15)	0.0235 (14)	0.0577 (19)	−0.0019 (12)	−0.0023 (14)	−0.0030 (13)
O37	0.066 (2)	0.049 (2)	0.0378 (18)	−0.0022 (17)	0.0053 (16)	−0.0092 (15)
O69	0.0301 (13)	0.0239 (13)	0.0309 (13)	−0.0014 (10)	−0.0020 (11)	−0.0035 (11)
O70	0.0267 (13)	0.0355 (15)	0.0346 (14)	−0.0032 (12)	0.0000 (11)	−0.0098 (12)
O71	0.0275 (13)	0.0274 (14)	0.0423 (15)	0.0023 (11)	0.0010 (12)	−0.0063 (12)
O72	0.0386 (16)	0.0436 (17)	0.0318 (15)	0.0011 (13)	0.0000 (12)	−0.0137 (13)
O73	0.0302 (13)	0.0231 (13)	0.0281 (13)	−0.0007 (10)	−0.0016 (11)	−0.0017 (10)
O74	0.0290 (13)	0.0271 (13)	0.0258 (12)	−0.0002 (10)	0.0031 (10)	−0.0004 (10)

O75	0.0353 (15)	0.0326 (15)	0.0337 (14)	−0.0034 (11)	0.0033 (12)	−0.0088 (12)
O76	0.0400 (16)	0.0517 (19)	0.0323 (15)	0.0006 (14)	−0.0018 (13)	0.0108 (14)
O77	0.0444 (17)	0.0240 (15)	0.0533 (18)	0.0019 (12)	0.0034 (14)	−0.0123 (13)
O94	0.058 (2)	0.168 (6)	0.045 (2)	−0.016 (3)	0.0077 (18)	−0.016 (3)

Geometric parameters (Å, º)

C1—C2	1.465 (5)	C43—C44	1.479 (7)
C1—C10	1.540 (6)	C44—H44A	0.9900
C1—O29	1.217 (5)	C44—H44B	0.9900
C2—H2	0.9500	C44—H44C	0.9900
C2—C3	1.335 (6)	C44—H44D	0.9900
C3—H3	0.9500	C44—C45A	1.512 (11)
C3—C4	1.473 (7)	C44—C45B	1.502 (14)
C4—H4AA	0.9900	C45A—C46A	1.316 (11)
C4—H4AB	0.9900	C45A—C50	1.530 (11)
C4—H4BC	0.9900	C46A—H46A	0.9500
C4—H4BD	0.9900	C46A—C47	1.484 (11)
C4—C5A	1.514 (8)	C45B—C46B	1.470 (14)
C4—C5B	1.499 (15)	C45B—O79	1.51 (7)
C5A—C6A	1.324 (8)	C45B—C50	1.527 (14)
C5A—C10	1.523 (8)	C46B—H46B	1.0000
C6A—H6A	0.9500	C46B—O79	1.51 (7)
C6A—C7	1.499 (8)	C46B—C47	1.493 (14)
C5B—C6B	1.472 (16)	C47—H47A	0.9900
C5B—O39	1.51 (8)	C47—H47B	0.9900
C5B—C10	1.522 (15)	C47—H47C	0.9900
C6B—H6B	1.0000	C47—H47D	0.9900
C6B—O39	1.51 (7)	C47—C48	1.535 (5)
C6B—C7	1.498 (16)	C48—H48	1.0000
C7—H7AA	0.9900	C48—C49	1.547 (5)
C7—H7AB	0.9900	C48—C61	1.525 (6)
C7—H7BC	0.9900	C49—H49	1.0000
C7—H7BD	0.9900	C49—C50	1.547 (5)
C7—C8	1.528 (6)	C49—C52	1.547 (5)
C8—H8	1.0000	C50—C51	1.554 (5)
C8—C9	1.546 (5)	C51—H51A	0.9800
C8—C21	1.529 (6)	C51—H51B	0.9800
C9—H9	1.0000	C51—H51C	0.9800
C9—C10	1.558 (5)	C52—H52A	0.9900
C9—C12	1.545 (5)	C52—H52B	0.9900
C10—C11	1.555 (5)	C52—C53	1.559 (5)
C11—H11A	0.9800	C53—H53A	0.9900
C11—H11B	0.9800	C53—H53B	0.9900
C11—H11C	0.9800	C53—C54	1.522 (5)
C12—H12A	0.9900	C54—C55	1.538 (5)
C12—H12B	0.9900	C54—C64	1.542 (5)
C12—C13	1.559 (5)	C54—O74	1.426 (5)
C13—H13A	0.9900	C55—O70	1.351 (5)
C13—H13B	0.9900	C55—O75	1.201 (5)

C13—C14	1.528 (5)	C56—C57	1.533 (6)
C14—C15	1.541 (5)	C56—C64	1.555 (5)
C14—C24	1.540 (5)	C56—C67	1.529 (6)
C14—O34	1.420 (4)	C56—O70	1.462 (5)
C15—O30	1.351 (5)	C57—H57	1.0000
C15—O35	1.195 (5)	C57—C66	1.498 (6)
C16—C17	1.539 (6)	C57—O71	1.448 (5)
C16—C24	1.546 (5)	C58—C59	1.530 (6)
C16—C27	1.525 (6)	C58—O71	1.333 (5)
C16—O30	1.458 (5)	C58—O77	1.199 (5)
C17—H17	1.0000	C59—H59	1.0000
C17—C26	1.508 (6)	C59—C60	1.537 (6)
C17—O31	1.452 (5)	C59—C65	1.559 (6)
C18—C19	1.528 (7)	C60—H60A	0.9900
C18—O31	1.343 (5)	C60—H60B	0.9900
C18—O37	1.196 (6)	C60—O72	1.454 (6)
C19—H19	1.0000	C61—C62	1.544 (6)
C19—C20	1.549 (6)	C61—O72	1.411 (5)
C19—C25	1.561 (6)	C61—O73	1.437 (5)
C20—H20A	0.9900	C62—C63	1.494 (6)
C20—H20B	0.9900	C62—O76	1.212 (5)
C20—O32	1.443 (5)	C63—H63	1.0000
C21—C22	1.528 (6)	C63—C64	1.541 (5)
C21—O32	1.408 (5)	C63—C65	1.555 (6)
C21—O33	1.445 (5)	C64—O73	1.420 (5)
C22—C23	1.504 (5)	C65—C66	1.524 (6)
C22—O36	1.204 (5)	C65—C68	1.526 (6)
C23—H23	1.0000	C66—H66A	0.9900
C23—C24	1.548 (5)	C66—H66B	0.9900
C23—C25	1.556 (6)	C67—H67A	0.9800
C24—O33	1.420 (4)	C67—H67B	0.9800
C25—C26	1.520 (6)	C67—H67C	0.9800
C25—C28	1.523 (6)	C68—H68A	0.9800
C26—H26A	0.9900	C68—H68B	0.9800
C26—H26B	0.9900	C68—H68C	0.9800
C27—H27A	0.9800	C91—H91A	0.9800
C27—H27B	0.9800	C91—H91B	0.9800
C27—H27C	0.9800	C91—H91C	0.9800
C28—H28A	0.9800	C91—C92	1.498 (8)
C28—H28B	0.9800	C92—C93	1.467 (8)
C28—H28C	0.9800	C92—O94	1.199 (6)
C41—C42	1.455 (6)	C93—H93A	0.9800
C41—C50	1.541 (6)	C93—H93B	0.9800
C41—O69	1.221 (5)	C93—H93C	0.9800
C42—H42	0.9500	O34—H34	0.8400
C42—C43	1.343 (6)	O74—H74	0.8400
C43—H43	0.9500		

C2—C1—C10	118.0 (3)	C43—C44—C45A	114.0 (15)

O29—C1—C2	119.9 (4)	C43—C44—C45B	116.7 (19)
O29—C1—C10	122.1 (3)	H44A—C44—H44B	107.6
C1—C2—H2	119.6	H44A—C44—H44D	102.9
C3—C2—C1	120.8 (4)	H44B—C44—H44C	112.0
C3—C2—H2	119.6	H44C—C44—H44D	107.3
C2—C3—H3	118.0	C45A—C44—H44A	108.8
C2—C3—C4	123.9 (4)	C45A—C44—H44B	108.8
C4—C3—H3	118.0	C45A—C44—H44C	105.3
C3—C4—H4AA	108.9	C45A—C44—H44D	113.6
C3—C4—H4AB	108.9	C45B—C44—H44A	111.3
C3—C4—H4BC	108.6	C45B—C44—H44B	103.2
C3—C4—H4BD	108.6	C45B—C44—H44C	108.1
C3—C4—C5A	113.5 (7)	C45B—C44—H44D	108.1
C3—C4—C5B	114 (2)	C44—C45A—C50	115.8 (10)
H4AA—C4—H4AB	107.7	C46A—C45A—C44	124.1 (9)
H4AA—C4—H4BD	105.9	C46A—C45A—C50	120.1 (12)
H4AB—C4—H4BC	109.4	C45A—C46A—H46A	116.1
H4BC—C4—H4BD	107.6	C45A—C46A—C47	127.8 (15)
C5A—C4—H4AA	108.9	C47—C46A—H46A	116.1
C5A—C4—H4AB	108.9	C44—C45B—O79	107 (4)
C5A—C4—H4BC	107.5	C44—C45B—C50	116.6 (12)
C5A—C4—H4BD	110.8	C46B—C45B—C44	119.4 (11)
C5B—C4—H4AA	110.0	C46B—C45B—O79	60.8 (17)
C5B—C4—H4AB	106.7	C46B—C45B—C50	122.4 (17)
C5B—C4—H4BC	108.6	O79—C45B—C50	113 (4)
C5B—C4—H4BD	108.6	C45B—C46B—H46B	118.5
C4—C5A—C10	116.3 (6)	C45B—C46B—O79	60.9 (18)
C6A—C5A—C4	122.3 (6)	C45B—C46B—C47	118.2 (19)
C6A—C5A—C10	121.4 (7)	O79—C46B—H46B	118.5
C5A—C6A—H6A	117.3	C47—C46B—H46B	118.5
C5A—C6A—C7	125.4 (8)	C47—C46B—O79	108 (4)
C7—C6A—H6A	117.3	C46B—O79—C45B	58 (3)
C4—C5B—O39	105 (4)	C46A—C47—H47A	109.5
C4—C5B—C10	117.3 (13)	C46A—C47—H47B	109.5
C6B—C5B—C4	118.9 (12)	C46A—C47—H47C	105.0
C6B—C5B—O39	60.7 (18)	C46A—C47—H47D	116.1
C6B—C5B—C10	123.1 (18)	C46A—C47—C48	110.7 (13)
O39—C5B—C10	112 (4)	C46B—C47—H47A	112.6
C5B—C6B—H6B	119.5	C46B—C47—H47B	102.0
C5B—C6B—O39	60.8 (18)	C46B—C47—H47C	108.6
C5B—C6B—C7	117 (2)	C46B—C47—H47D	108.6
O39—C6B—H6B	119.5	C46B—C47—C48	114.7 (18)
C7—C6B—H6B	119.5	H47A—C47—H47B	108.1
C7—C6B—O39	106 (4)	H47A—C47—H47D	102.0
C6B—O39—C5B	59 (3)	H47B—C47—H47C	113.5
C6A—C7—H7AA	109.4	H47C—C47—H47D	107.6
C6A—C7—H7AB	109.4	C48—C47—H47A	109.5
C6A—C7—H7BC	108.5	C48—C47—H47B	109.5
C6A—C7—H7BD	113.2	C48—C47—H47C	108.6

C6A—C7—C8	111.0 (6)	C48—C47—H47D	108.6
C6B—C7—H7AA	109.0	C47—C48—H48	108.2
C6B—C7—H7AB	104.5	C47—C48—C49	108.9 (3)
C6B—C7—H7BC	108.3	C49—C48—H48	108.2
C6B—C7—H7BD	108.3	C61—C48—C47	108.7 (3)
C6B—C7—C8	116.1 (18)	C61—C48—H48	108.2
H7AA—C7—H7AB	108.0	C61—C48—C49	114.3 (3)
H7AA—C7—H7BD	105.3	C48—C49—H49	107.0
H7AB—C7—H7BC	110.2	C48—C49—C50	111.7 (3)
H7BC—C7—H7BD	107.4	C48—C49—C52	113.5 (3)
C8—C7—H7AA	109.4	C50—C49—H49	107.0
C8—C7—H7AB	109.4	C52—C49—H49	107.0
C8—C7—H7BC	108.3	C52—C49—C50	110.2 (3)
C8—C7—H7BD	108.3	C41—C50—C49	107.7 (3)
C7—C8—H8	108.2	C41—C50—C51	107.7 (3)
C7—C8—C9	109.2 (3)	C45A—C50—C41	106.6 (19)
C7—C8—C21	108.3 (3)	C45A—C50—C49	112.9 (8)
C9—C8—H8	108.2	C45A—C50—C51	108 (2)
C21—C8—H8	108.2	C45B—C50—C41	110 (3)
C21—C8—C9	114.5 (3)	C45B—C50—C49	114.6 (11)
C8—C9—H9	106.7	C45B—C50—C51	103 (3)
C8—C9—C10	110.8 (3)	C49—C50—C51	113.3 (3)
C10—C9—H9	106.7	C50—C51—H51A	109.5
C12—C9—C8	115.6 (3)	C50—C51—H51B	109.5
C12—C9—H9	106.7	C50—C51—H51C	109.5
C12—C9—C10	109.9 (3)	H51A—C51—H51B	109.5
C1—C10—C9	107.1 (3)	H51A—C51—H51C	109.5
C1—C10—C11	108.6 (3)	H51B—C51—H51C	109.5
C5A—C10—C1	106.4 (9)	C49—C52—H52A	107.4
C5A—C10—C9	113.6 (5)	C49—C52—H52B	107.4
C5A—C10—C11	107.7 (11)	C49—C52—C53	119.9 (3)
C5B—C10—C1	108 (3)	H52A—C52—H52B	106.9
C5B—C10—C9	114.7 (11)	C53—C52—H52A	107.4
C5B—C10—C11	105 (3)	C53—C52—H52B	107.4
C11—C10—C9	113.1 (3)	C52—C53—H53A	107.4
C10—C11—H11A	109.5	C52—C53—H53B	107.4
C10—C11—H11B	109.5	H53A—C53—H53B	106.9
C10—C11—H11C	109.5	C54—C53—C52	119.6 (3)
H11A—C11—H11B	109.5	C54—C53—H53A	107.4
H11A—C11—H11C	109.5	C54—C53—H53B	107.4
H11B—C11—H11C	109.5	C53—C54—C55	111.2 (3)
C9—C12—H12A	107.1	C53—C54—C64	120.1 (3)
C9—C12—H12B	107.1	C55—C54—C64	99.2 (3)
C9—C12—C13	120.9 (3)	O74—C54—C53	111.3 (3)
H12A—C12—H12B	106.8	O74—C54—C55	106.1 (3)
C13—C12—H12A	107.1	O74—C54—C64	107.6 (3)
C13—C12—H12B	107.1	O70—C55—C54	109.6 (3)
C12—C13—H13A	107.3	O75—C55—C54	128.8 (4)
C12—C13—H13B	107.3	O75—C55—O70	121.5 (3)

H13A—C13—H13B	106.9	C57—C56—C64	114.4 (3)
C14—C13—C12	120.0 (3)	C67—C56—C57	109.5 (3)
C14—C13—H13A	107.3	C67—C56—C64	115.6 (3)
C14—C13—H13B	107.3	O70—C56—C57	105.3 (3)
C13—C14—C15	111.7 (3)	O70—C56—C64	101.8 (3)
C13—C14—C24	120.1 (3)	O70—C56—C67	109.3 (3)
C24—C14—C15	99.1 (3)	C56—C57—H57	107.6
O34—C14—C13	111.0 (3)	C66—C57—C56	114.8 (3)
O34—C14—C15	106.3 (3)	C66—C57—H57	107.6
O34—C14—C24	107.3 (3)	O71—C57—C56	107.6 (3)
O30—C15—C14	109.7 (3)	O71—C57—H57	107.6
O35—C15—C14	128.4 (4)	O71—C57—C66	111.4 (3)
O35—C15—O30	121.8 (3)	O71—C58—C59	119.3 (3)
C17—C16—C24	114.8 (3)	O77—C58—C59	121.5 (4)
C27—C16—C17	108.9 (3)	O77—C58—O71	118.9 (4)
C27—C16—C24	115.8 (3)	C58—C59—H59	103.3
O30—C16—C17	105.2 (3)	C58—C59—C60	109.1 (4)
O30—C16—C24	102.0 (3)	C58—C59—C65	117.4 (3)
O30—C16—C27	109.4 (3)	C60—C59—H59	103.3
C16—C17—H17	107.8	C60—C59—C65	117.9 (3)
C26—C17—C16	114.1 (3)	C65—C59—H59	103.3
C26—C17—H17	107.8	C59—C60—H60A	107.8
O31—C17—C16	107.9 (3)	C59—C60—H60B	107.8
O31—C17—H17	107.8	H60A—C60—H60B	107.1
O31—C17—C26	111.1 (3)	O72—C60—C59	118.0 (4)
O31—C18—C19	119.1 (4)	O72—C60—H60A	107.8
O37—C18—C19	122.0 (4)	O72—C60—H60B	107.8
O37—C18—O31	118.6 (4)	C48—C61—C62	116.7 (3)
C18—C19—H19	102.9	O72—C61—C48	109.0 (3)
C18—C19—C20	109.6 (4)	O72—C61—C62	106.6 (3)
C18—C19—C25	117.8 (3)	O72—C61—O73	108.7 (3)
C20—C19—H19	102.9	O73—C61—C48	110.5 (3)
C20—C19—C25	118.1 (4)	O73—C61—C62	105.0 (3)
C25—C19—H19	102.9	C63—C62—C61	106.5 (3)
C19—C20—H20A	107.9	O76—C62—C61	124.9 (4)
C19—C20—H20B	107.9	O76—C62—C63	128.3 (4)
H20A—C20—H20B	107.2	C62—C63—H63	111.0
O32—C20—C19	117.8 (3)	C62—C63—C64	101.3 (3)
O32—C20—H20A	107.9	C62—C63—C65	107.5 (3)
O32—C20—H20B	107.9	C64—C63—H63	111.0
C22—C21—C8	116.7 (3)	C64—C63—C65	114.5 (3)
O32—C21—C8	108.3 (3)	C65—C63—H63	111.0
O32—C21—C22	108.9 (3)	C54—C64—C56	101.9 (3)
O32—C21—O33	107.5 (3)	C63—C64—C54	109.6 (3)
O33—C21—C8	110.6 (3)	C63—C64—C56	114.6 (3)
O33—C21—C22	104.5 (3)	O73—C64—C54	114.7 (3)
C23—C22—C21	107.1 (3)	O73—C64—C56	110.9 (3)
O36—C22—C21	124.9 (4)	O73—C64—C63	105.5 (3)
O36—C22—C23	127.7 (4)	C63—C65—C59	114.5 (3)

C22—C23—H23	110.9	C66—C65—C59	106.8 (3)
C22—C23—C24	100.1 (3)	C66—C65—C63	106.9 (3)
C22—C23—C25	108.8 (3)	C66—C65—C68	109.5 (3)
C24—C23—H23	110.9	C68—C65—C59	110.5 (3)
C24—C23—C25	114.7 (3)	C68—C65—C63	108.6 (3)
C25—C23—H23	110.9	C57—C66—C65	109.7 (3)
C14—C24—C16	102.1 (3)	C57—C66—H66A	109.7
C14—C24—C23	109.6 (3)	C57—C66—H66B	109.7
C16—C24—C23	114.1 (3)	C65—C66—H66A	109.7
O33—C24—C14	115.1 (3)	C65—C66—H66B	109.7
O33—C24—C16	111.3 (3)	H66A—C66—H66B	108.2
O33—C24—C23	104.9 (3)	C56—C67—H67A	109.5
C23—C25—C19	114.2 (3)	C56—C67—H67B	109.5
C26—C25—C19	107.6 (4)	C56—C67—H67C	109.5
C26—C25—C23	106.5 (3)	H67A—C67—H67B	109.5
C26—C25—C28	110.2 (4)	H67A—C67—H67C	109.5
C28—C25—C19	109.5 (4)	H67B—C67—H67C	109.5
C28—C25—C23	108.7 (4)	C65—C68—H68A	109.5
C17—C26—C25	109.8 (3)	C65—C68—H68B	109.5
C17—C26—H26A	109.7	C65—C68—H68C	109.5
C17—C26—H26B	109.7	H68A—C68—H68B	109.5
C25—C26—H26A	109.7	H68A—C68—H68C	109.5
C25—C26—H26B	109.7	H68B—C68—H68C	109.5
H26A—C26—H26B	108.2	H91A—C91—H91B	109.5
C16—C27—H27A	109.5	H91A—C91—H91C	109.5
C16—C27—H27B	109.5	H91B—C91—H91C	109.5
C16—C27—H27C	109.5	C92—C91—H91A	109.5
H27A—C27—H27B	109.5	C92—C91—H91B	109.5
H27A—C27—H27C	109.5	C92—C91—H91C	109.5
H27B—C27—H27C	109.5	C93—C92—C91	116.8 (4)
C25—C28—H28A	109.5	O94—C92—C91	122.0 (5)
C25—C28—H28B	109.5	O94—C92—C93	121.2 (6)
C25—C28—H28C	109.5	C92—C93—H93A	109.5
H28A—C28—H28B	109.5	C92—C93—H93B	109.5
H28A—C28—H28C	109.5	C92—C93—H93C	109.5
H28B—C28—H28C	109.5	H93A—C93—H93B	109.5
C42—C41—C50	118.3 (3)	H93A—C93—H93C	109.5
O69—C41—C42	120.9 (4)	H93B—C93—H93C	109.5
O69—C41—C50	120.9 (3)	C15—O30—C16	111.2 (3)
C41—C42—H42	119.4	C18—O31—C17	121.4 (3)
C43—C42—C41	121.2 (4)	C21—O32—C20	117.9 (3)
C43—C42—H42	119.4	C24—O33—C21	110.4 (3)
C42—C43—H43	118.4	C14—O34—H34	109.5
C42—C43—C44	123.2 (4)	C55—O70—C56	111.4 (3)
C44—C43—H43	118.4	C58—O71—C57	120.4 (3)
C43—C44—H44A	108.8	C61—O72—C60	118.5 (3)
C43—C44—H44B	108.8	C64—O73—C61	110.2 (3)
C43—C44—H44C	108.1	C54—O74—H74	109.5
C43—C44—H44D	108.1		

C1—C2—C3—C4	2.3 (7)	C46B—C45B—C50—C41	124 (2)
C2—C1—C10—C5A	42.7 (9)	C46B—C45B—C50—C49	3 (5)
C2—C1—C10—C5B	41 (3)	C46B—C45B—C50—C51	−121 (3)
C2—C1—C10—C9	164.6 (3)	C46B—C47—C48—C49	−55 (3)
C2—C1—C10—C11	−72.9 (4)	C46B—C47—C48—C61	180 (3)
C2—C3—C4—C5A	−10.8 (12)	O79—C45B—C46B—C47	−96 (4)
C2—C3—C4—C5B	−9 (3)	O79—C45B—C50—C41	−167 (2)
C3—C4—C5A—C6A	−139.1 (10)	O79—C45B—C50—C49	72 (5)
C3—C4—C5A—C10	38 (2)	O79—C45B—C50—C51	−52 (3)
C3—C4—C5B—C6B	−137 (2)	O79—C46B—C47—C48	−43 (3)
C3—C4—C5B—O39	158.4 (16)	C47—C46B—O79—C45B	113 (2)
C3—C4—C5B—C10	34 (7)	C47—C48—C49—C50	60.7 (4)
C4—C5A—C6A—C7	178 (2)	C47—C48—C49—C52	−174.0 (3)
C4—C5A—C10—C1	−52 (2)	C47—C48—C61—C62	−172.8 (3)
C4—C5A—C10—C9	−169.5 (14)	C47—C48—C61—O72	−52.1 (4)
C4—C5A—C10—C11	64 (2)	C47—C48—C61—O73	67.3 (4)
C4—C5B—C6B—O39	−92 (4)	C48—C49—C50—C41	−158.1 (3)
C4—C5B—C6B—C7	175 (8)	C48—C49—C50—C45A	−41 (3)
C4—C5B—O39—C6B	115 (3)	C48—C49—C50—C45B	−35 (4)
C4—C5B—C10—C1	−48 (7)	C48—C49—C50—C51	82.8 (4)
C4—C5B—C10—C9	−167 (4)	C48—C49—C52—C53	97.9 (4)
C4—C5B—C10—C11	68 (6)	C48—C61—C62—C63	−134.2 (3)
C5A—C6A—C7—C8	21.6 (13)	C48—C61—C62—O76	52.2 (5)
C6A—C5A—C10—C1	125.1 (11)	C48—C61—O72—C60	−157.6 (3)
C6A—C5A—C10—C5B	−112 (42)	C48—C61—O73—C64	115.5 (3)
C6A—C5A—C10—C9	7.5 (17)	C49—C48—C61—C62	65.3 (4)
C6A—C5A—C10—C11	−118.6 (12)	C49—C48—C61—O72	−174.0 (3)
C6A—C7—C8—C9	−51.3 (10)	C49—C48—C61—O73	−54.6 (4)
C6A—C7—C8—C21	−176.6 (10)	C49—C52—C53—C54	−19.3 (5)
C5B—C6B—C7—C8	21 (3)	C50—C41—C42—C43	−18.9 (6)
C6B—C5B—C10—C1	122 (3)	C50—C45A—C46A—C47	1 (3)
C6B—C5B—C10—C9	3 (5)	C50—C45B—C46B—O79	100 (5)
C6B—C5B—C10—C11	−122 (3)	C50—C45B—C46B—C47	4 (3)
C6B—C7—C8—C9	−54 (3)	C50—C45B—O79—C46B	−115 (4)
C6B—C7—C8—C21	−179 (3)	C50—C49—C52—C53	−136.0 (3)
O39—C5B—C6B—C7	−93 (4)	C52—C49—C50—C41	74.8 (4)
O39—C5B—C10—C1	−169 (2)	C52—C49—C50—C45A	−168 (3)
O39—C5B—C10—C9	72 (5)	C52—C49—C50—C45B	−162 (4)
O39—C5B—C10—C11	−53 (3)	C52—C49—C50—C51	−44.3 (4)
O39—C6B—C7—C8	−43 (3)	C52—C53—C54—C55	−175.2 (3)
C7—C6B—O39—C5B	113 (2)	C52—C53—C54—C64	−60.2 (5)
C7—C8—C9—C10	60.7 (4)	C52—C53—C54—O74	66.8 (4)
C7—C8—C9—C12	−173.4 (3)	C53—C54—C55—O70	154.0 (3)
C7—C8—C21—C22	−173.7 (3)	C53—C54—C55—O75	−29.0 (6)
C7—C8—C21—O32	−50.5 (4)	C53—C54—C64—C56	−158.6 (3)
C7—C8—C21—O33	67.1 (4)	C53—C54—C64—C63	−36.8 (4)
C8—C9—C10—C1	−155.4 (3)	C53—C54—C64—O73	81.5 (4)
C8—C9—C10—C5A	−38.2 (13)	C54—C55—O70—C56	−3.4 (4)

C8—C9—C10—C5B	−36 (4)	C54—C64—O73—C61	−91.8 (4)
C8—C9—C10—C11	85.0 (4)	C55—C54—C64—C56	−37.5 (3)
C8—C9—C12—C13	92.8 (4)	C55—C54—C64—C63	84.3 (3)
C8—C21—C22—C23	−135.7 (3)	C55—C54—C64—O73	−157.4 (3)
C8—C21—C22—O36	49.3 (5)	C56—C57—C66—C65	59.2 (4)
C8—C21—O32—C20	−156.9 (4)	C56—C57—O71—C58	−83.4 (4)
C8—C21—O33—C24	115.5 (3)	C56—C64—O73—C61	153.4 (3)
C9—C8—C21—C22	64.2 (4)	C57—C56—C64—C54	149.8 (3)
C9—C8—C21—O32	−172.5 (3)	C57—C56—C64—C63	31.6 (5)
C9—C8—C21—O33	−54.9 (5)	C57—C56—C64—O73	−87.7 (4)
C9—C12—C13—C14	−12.5 (5)	C57—C56—O70—C55	−140.9 (3)
C10—C1—C2—C3	−20.1 (6)	C58—C59—C60—O72	−60.1 (5)
C10—C5A—C6A—C7	1.2 (15)	C58—C59—C65—C63	82.4 (4)
C10—C5B—C6B—O39	98 (5)	C58—C59—C65—C66	−35.6 (5)
C10—C5B—C6B—C7	5 (4)	C58—C59—C65—C68	−154.6 (4)
C10—C5B—O39—C6B	−117 (4)	C59—C58—O71—C57	−20.0 (5)
C10—C9—C12—C13	−140.8 (3)	C59—C60—O72—C61	−46.6 (5)
C12—C9—C10—C1	75.5 (4)	C59—C65—C66—C57	58.0 (4)
C12—C9—C10—C5A	−167.3 (12)	C60—C59—C65—C63	−51.4 (5)
C12—C9—C10—C5B	−165 (4)	C60—C59—C65—C66	−169.5 (4)
C12—C9—C10—C11	−44.1 (4)	C60—C59—C65—C68	71.6 (5)
C12—C13—C14—C15	−179.1 (3)	C61—C48—C49—C50	−177.5 (3)
C12—C13—C14—C24	−63.7 (5)	C61—C48—C49—C52	−52.2 (4)
C12—C13—C14—O34	62.5 (4)	C61—C62—C63—C64	27.1 (4)
C13—C14—C15—O30	152.7 (3)	C61—C62—C63—C65	−93.2 (4)
C13—C14—C15—O35	−30.6 (5)	C62—C61—O72—C60	−30.8 (5)
C13—C14—C24—C16	−158.7 (3)	C62—C61—O73—C64	−11.1 (4)
C13—C14—C24—C23	−37.3 (4)	C62—C63—C64—C54	89.7 (4)
C13—C14—C24—O33	80.6 (4)	C62—C63—C64—C56	−156.4 (3)
C14—C15—O30—C16	−1.7 (4)	C62—C63—C64—O73	−34.2 (4)
C14—C24—O33—C21	−90.6 (4)	C62—C63—C65—C59	50.8 (4)
C15—C14—C24—C16	−36.9 (3)	C62—C63—C65—C66	168.8 (3)
C15—C14—C24—C23	84.4 (3)	C62—C63—C65—C68	−73.2 (4)
C15—C14—C24—O33	−157.6 (3)	C63—C64—O73—C61	28.9 (4)
C16—C17—C26—C25	59.7 (5)	C63—C65—C66—C57	−65.0 (4)
C16—C17—O31—C18	−84.8 (4)	C64—C54—C55—O70	26.6 (4)
C16—C24—O33—C21	153.8 (3)	C64—C54—C55—O75	−156.4 (4)
C17—C16—C24—C14	150.4 (3)	C64—C56—C57—C66	−41.2 (5)
C17—C16—C24—C23	32.2 (5)	C64—C56—C57—O71	83.4 (4)
C17—C16—C24—O33	−86.3 (4)	C64—C56—O70—C55	−21.2 (4)
C17—C16—O30—C15	−142.7 (3)	C64—C63—C65—C59	−60.9 (4)
C18—C19—C20—O32	−60.7 (5)	C64—C63—C65—C66	57.1 (4)
C18—C19—C25—C23	84.6 (5)	C64—C63—C65—C68	175.2 (3)
C18—C19—C25—C26	−33.4 (5)	C65—C59—C60—O72	77.2 (5)
C18—C19—C25—C28	−153.3 (4)	C65—C63—C64—C54	−155.0 (3)
C19—C18—O31—C17	−16.0 (5)	C65—C63—C64—C56	−41.1 (4)
C19—C20—O32—C21	−48.7 (6)	C65—C63—C64—O73	81.1 (4)
C19—C25—C26—C17	57.5 (4)	C66—C57—O71—C58	43.3 (5)
C20—C19—C25—C23	−50.8 (5)	C67—C56—C57—C66	−172.8 (3)

C20—C19—C25—C26	−168.8 (4)	C67—C56—C57—O71	−48.2 (4)
C20—C19—C25—C28	71.3 (5)	C67—C56—C64—C54	−81.6 (4)
C21—C8—C9—C10	−177.8 (3)	C67—C56—C64—C63	160.1 (3)
C21—C8—C9—C12	−51.9 (5)	C67—C56—C64—O73	40.9 (5)
C21—C22—C23—C24	29.4 (4)	C67—C56—O70—C55	101.5 (4)
C21—C22—C23—C25	−91.3 (4)	C68—C65—C66—C57	177.6 (3)
C22—C21—O32—C20	−29.1 (5)	O29—C1—C2—C3	158.1 (4)
C22—C21—O33—C24	−10.8 (4)	O29—C1—C10—C5A	−135.4 (9)
C22—C23—C24—C14	88.3 (3)	O29—C1—C10—C5B	−137 (3)
C22—C23—C24—C16	−157.9 (3)	O29—C1—C10—C9	−13.5 (5)
C22—C23—C24—O33	−35.9 (4)	O29—C1—C10—C11	108.9 (4)
C22—C23—C25—C19	50.1 (4)	O30—C16—C17—C26	69.4 (4)
C22—C23—C25—C26	168.7 (3)	O30—C16—C17—O31	−166.5 (3)
C22—C23—C25—C28	−72.5 (4)	O30—C16—C24—C14	37.2 (3)
C23—C24—O33—C21	30.0 (4)	O30—C16—C24—C23	−80.9 (4)
C23—C25—C26—C17	−65.3 (4)	O30—C16—C24—O33	160.6 (3)
C24—C14—C15—O30	25.1 (3)	O31—C17—C26—C25	−62.7 (4)
C24—C14—C15—O35	−158.2 (4)	O31—C18—C19—C20	151.5 (4)
C24—C16—C17—C26	−41.8 (5)	O31—C18—C19—C25	12.6 (6)
C24—C16—C17—O31	82.2 (4)	O32—C21—C22—C23	101.4 (4)
C24—C16—O30—C15	−22.6 (4)	O32—C21—C22—O36	−73.6 (5)
C24—C23—C25—C19	−61.1 (4)	O32—C21—O33—C24	−126.5 (3)
C24—C23—C25—C26	57.5 (4)	O33—C21—C22—C23	−13.3 (4)
C24—C23—C25—C28	176.3 (3)	O33—C21—C22—O36	171.7 (4)
C25—C19—C20—O32	78.0 (5)	O33—C21—O32—C20	83.6 (4)
C25—C23—C24—C14	−155.4 (3)	O34—C14—C15—O30	−86.1 (3)
C25—C23—C24—C16	−41.6 (5)	O34—C14—C15—O35	90.6 (4)
C25—C23—C24—O33	80.4 (4)	O34—C14—C24—C16	73.4 (3)
C26—C17—O31—C18	41.0 (5)	O34—C14—C24—C23	−165.2 (3)
C27—C16—C17—C26	−173.4 (3)	O34—C14—C24—O33	−47.3 (4)
C27—C16—C17—O31	−49.4 (4)	O35—C15—O30—C16	−178.7 (3)
C27—C16—C24—C14	−81.4 (4)	O36—C22—C23—C24	−155.7 (4)
C27—C16—C24—C23	160.4 (3)	O36—C22—C23—C25	83.6 (5)
C27—C16—C24—O33	42.0 (4)	O37—C18—C19—C20	−35.2 (6)
C27—C16—O30—C15	100.5 (4)	O37—C18—C19—C25	−174.1 (4)
C28—C25—C26—C17	176.9 (4)	O37—C18—O31—C17	170.5 (4)
C41—C42—C43—C44	1.1 (7)	O69—C41—C42—C43	161.8 (4)
C42—C41—C50—C45A	42.2 (19)	O69—C41—C50—C45A	−138.4 (19)
C42—C41—C50—C45B	38 (3)	O69—C41—C50—C45B	−143 (3)
C42—C41—C50—C49	163.7 (3)	O69—C41—C50—C49	−17.0 (5)
C42—C41—C50—C51	−73.7 (4)	O69—C41—C50—C51	105.6 (4)
C42—C43—C44—C45A	−10 (3)	O70—C56—C57—C66	69.8 (4)
C42—C43—C44—C45B	−5 (4)	O70—C56—C57—O71	−165.6 (3)
C43—C44—C45A—C46A	−141 (2)	O70—C56—C64—C54	36.7 (4)
C43—C44—C45A—C50	38 (5)	O70—C56—C64—C63	−81.5 (4)
C43—C44—C45B—C46B	−139.5 (18)	O70—C56—C64—O73	159.3 (3)
C43—C44—C45B—O79	154.8 (14)	O71—C57—C66—C65	−63.4 (4)
C43—C44—C45B—C50	27 (7)	O71—C58—C59—C60	154.6 (3)
C44—C45A—C46A—C47	−179 (6)	O71—C58—C59—C65	17.0 (5)

C44—C45A—C50—C41	−52 (5)	O72—C61—C62—C63	103.8 (4)
C44—C45A—C50—C45B	81 (10)	O72—C61—C62—O76	−69.7 (5)
C44—C45A—C50—C49	−170 (3)	O72—C61—O73—C64	−124.9 (3)
C44—C45A—C50—C51	64 (4)	O73—C61—C62—C63	−11.5 (4)
C44—C45B—C46B—O79	−94 (4)	O73—C61—C62—O76	175.0 (4)
C44—C45B—C46B—C47	169 (7)	O73—C61—O72—C60	81.9 (4)
C44—C45B—O79—C46B	115 (3)	O74—C54—C55—O70	−84.9 (4)
C44—C45B—C50—C41	−42 (7)	O74—C54—C55—O75	92.2 (5)
C44—C45B—C50—C49	−163 (4)	O74—C54—C64—C56	72.8 (3)
C44—C45B—C50—C51	73 (6)	O74—C54—C64—C63	−165.5 (3)
C45A—C46A—C47—C48	19 (3)	O74—C54—C64—O73	−47.1 (4)
C46A—C45A—C50—C41	128 (2)	O75—C55—O70—C56	179.3 (4)
C46A—C45A—C50—C49	10 (4)	O76—C62—C63—C64	−159.6 (4)
C46A—C45A—C50—C51	−117 (2)	O76—C62—C63—C65	80.0 (5)
C46A—C47—C48—C49	−48 (2)	O77—C58—C59—C60	−31.6 (5)
C46A—C47—C48—C61	−173 (2)	O77—C58—C59—C65	−169.2 (4)
C45B—C46B—C47—C48	23 (3)	O77—C58—O71—C57	166.0 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O34—H34···O69	0.84	1.91	2.739 (4)	172
O74—H74···O29	0.84	2.00	2.801 (4)	160
C52—H52*A*···O76	0.99	2.38	3.217 (5)	142
C17—H17···O75^{i}	1.00	2.59	3.415 (5)	140
C53—H53*B*···O77^{ii}	0.99	2.49	3.453 (5)	163
C63—H63···O77^{ii}	1.00	2.39	3.376 (5)	170
C66—H66*B*···O94^{iii}	0.99	2.58	3.457 (6)	148
C91—H91*C*···O37^{iv}	0.98	2.58	3.432 (7)	145

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, *y*−1/2, −*z*+2; (iii) −*x*+1, *y*+1/2, −*z*+2; (iv) −*x*+2, *y*−1/2, −*z*+1.

In vitro IC_{50} values (µg ml^{-1}) against Plasmodium falciparum (3D7) and against WI-38 cells, and selectivity index (SI)

Extracts	IC_{50} (3D7)	IC_{50} (WI-38)	SI
CH_2Cl_2	1.41±0.21	6.97±2.02	4.94
EtOH/H_2O (50:50 *v*/*v*)	9.05±0.87	36.48±4.41	4.02
H_2O	11.36±1.84	56.43±1.85	4.96
MeOH	3.16±1.13	13.95±3.68	4.41
(**X**)	0.86±0.13	3.13±0.81	3.63
(**Y**)	0.62±0.35	1.59±0.27	2.25
Chloroquine	0.0022±0.005		ND
Artemisinin	0.0047±1.39		ND
Camptothecine		13.84±3.82	ND